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### THERMAL CONDUCTIVITY AND VOLUMETRIC SPECIFIC HEAT MEASUREMENTS OF AN RTV-655/POLYIMIDE AEROGEL COMPOUND UNDER VARYING TEMPERATURE

by

Ken Marcus Mitchell

A Thesis

Submitted in Partial Fulfillment of the

Requirements for the Degree of

Master of Science

Major: Mechanical Engineering

The University of Memphis

August 2019

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# DEDICATION

I dedicate this work to my late father, Scott A. Mitchell, and late grandfather, Marcus J. Mitchell.

# ACKNOWLEDGEMENTS

I would like to acknowledge the Tennessee Space Grant, Dr. Jeffrey Marchetta, and Dr. Firouzeh Sabri for giving me the opportunity to work on this research and introducing me to space research.

#### ABSTRACT

Mitchell, Ken Marcus, M.S. The University of Memphis. May 2018. Thermal Conductivity and Volumetric Specific Heat Measurements of an RTV-655/Polyimide Aerogel Compound Under Varying Temperature. Major Professor: Jeffrey G. Marchetta, Ph.D.

The ability to store cryogenic liquids for long duration space missions is essential to future manned space exploration. Boil-off of cryogens due to incident solar radiation leads to self-pressurization of a cryogenic liquid tank. An ideal tank construction material would have low thermal conductivity and would retain its structural integrity through extreme temperatures. In previous research, a small-scale RTV-655/polyimide aerogel cryogenic liquid storage tank was constructed and tested to assess the performance of the compound material. Further development of RTV-655/polyimide aerogel cryogenic tanks for space applications is contingent upon performing computational studies to optimize the tank design and minimize costly experiments. Moreover, computational heat transfer models, specifically models simulating conduction heat transfer through the RTV-655/polyimide aerogel compound, are dependent on accurate, measured thermal conductivity and volumetric specific heat values for the RTV-655/polyimide aerogel compound at the temperatures of interest. Thermal conductivity and volumetric specific heat values of the combination of RTV-655 and polyimide aerogel have not been published at cryogenic temperatures. The transient plane source method was used to measure the thermal conductivity and volumetric specific heat for RTV-655, polyimide aerogel, and three volume ratios of the compound at 313K, 295K, 253K, and the cryogenic temperature of 85K.

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### Nomenclature

- α thermal diffusivity
- °C degree Celsius
- *C* specific heat
- $D(\tau)$  dimensionless geometric time dependent function
- *k* effective thermal conductivity
- $k_a$  axial thermal conductivity
- $k_r$  radial thermal conductivity
- K degree kelvin
- M sample mass
- mm millimeter
- *P<sub>ref</sub>* reference measurement power
- *P<sub>sam</sub>* sample measurement power
- *r* sensor radius
- $R_0$  initial sensor resistance
- R(t) time-dependent resistance of a sensor
- RTV room temperature vulcanizing rubber
- $\rho$  mass density
- $\rho C$  volumetric specific heat
- $t_{RTV}$  thickness of RTV-655
- *t*<sub>PI</sub> thickness of polyimide aerogel
- *TCR* temperature coefficient of electrical resistivity
- au total to characteristic time ratio

- $\Theta$  characteristic time
- *V*<sub>PI</sub> polyimide aerogel volume
- $V_{RTV}$  RTV volume
- VR sample volume ratio

#### CHAPTER 1.

#### INTRODUCTION AND BACKGROUND

#### 1.1 Introduction

Modern technologies for storing cryogenic liquids may be insufficient for future long-duration manned space exploration. Cryogenic liquids are liquefied gases which have very low boiling points, generally 120 Kelvin or below [1]. They have several applications in space including fueling chemical propulsion systems, supporting life support systems, and cooling critical components. The difficulty of storing cryogenic liquids for long durations is that any heat load experienced by the tank can quickly vaporize the liquid causing tank self-pressurization [2]. Thermodynamic vent systems and cryocoolers are examples of active methods to reduce self-pressurization while examples of passive methods include radiation shields and multi-layer insulation, MLI. MLI, the focus of this study, is usually the largest heat leak in cryogenic systems, so improvements in its thermal performance are desirable, especially for long-duration exploration [3]. Another design consideration for a space-based cryogenic tanks is the thermal cycling experienced in space due to variations in solar radiation. Modern storage tanks constructed of metals and composites are susceptible to thermal fatigue in space resulting in brittleness and micro-cracking [4]. A cryogenic tank can also benefit from a weight reduction, which subsequently reduces the launch vehicle mass. Combining all the design considerations, a material with a high yield strength, low thermal conductivity, low density, high fatigue strength, and durability through an extreme temperature range is ideal for a space-based cryogenic tank. An elastomer/aerogel compound was proposed as

a potential solution for increasing thermal resistance and thermal fatigue life of cryogenic liquid storage tanks [4]. Compared to metals and composites, elastomers have relatively low thermal conductivity while maintaining their structural integrity over a large temperature range. Aerogel is the primary insulator and weight reduction component. The elastomer/aerogel compound will provide structural support and is also an insulator. The elastomer and aerogel were chosen from previous research as RTV-655 and polyimide aerogel. RTV-655 is a low thermal conductivity, space-qualified silicone with great elastomeric properties. Polyimide aerogel is an aerogel with improved elasticity while maintaining its low thermal conductivity. Embedding polyimide aerogel in the walls of an RTV-655 tank may protect the aerogel, reduce the weight of the tank, and increase the thermal performance of the tank.

The purpose of this research is to measure the thermal conductivity and volumetric specific heat in relation to the volume ratio of polyimide aerogel and under varying temperature. Thermal conductivity and volumetric specific heat values for RTV-655, polyimide aerogel, and different volume ratios of the compound are necessary for design and optimization of an RTV-655/polyimide aerogel cryogenic liquid storage tank. Measured values of thermal conductivity and volumetric specific heat for each material, individually and as a multi-layer compound, would fill a gap in existing knowledge. The temperature range of interest is approximately 116-394K, the temperatures of the International Space Station on the dark side and sun side of Earth [5].

#### 1.2 Background

The idea of combining RTV-655 with polyimide aerogel originated from previous designs for insulating cryogenic tanks with an aerogel embedded in a "blanket" [6].

Polyimide aerogel was chosen for its mechanical strength and availability, and RTV-655 was chosen for its thermal resistance, elastomeric properties, moldability, and chemical resistance. Accurately measuring material properties for the compound at different volume ratios and at different temperatures of interest can reduce the complexity of computational models by consolidating a compound material into a single material property [7].

RTV-655, chemically characterized as a form of polydimethylsiloxane or PDMS, is a room temperature vulcanizing silicone rubber with a low thermal conductivity. RTV-655 exhibits elastomeric behavior throughout a wide temperature range and is transparent in the visible light spectrum. RTV-655 begins as a two-part compound that is mixed at a 10:1 mass ratio before being molded then cured. Therefore, RTV-655 can be molded into a variety of geometries. During the mixing process, air is introduced during mixing and must be outgassed in a vacuum chamber before being poured into a mold, of virtually any geometry, then outgassed again to eliminate any air bubbles introduced in the pouring process. The cure rate of RTV-655 can be accelerated by an increase in temperature for which temperature and times are documented by the manufacturer [8].

RTV-655 was chosen for its elastomeric behavior, low thermal conductivity, low glass transition temperature, and more importantly, RTV-655 is desirable for its low-outgassing behavior, within NASA requirements specified by ASTM E595 [9]. In comparison to RTV-655, composites and metals have a higher thermal conductivity and may be susceptible to micro-cracks and brittleness in space at extreme temperatures for long durations. At room temperature, RTV-655 has a low thermal conductivity of 0.17 W/m\*K, volumetric specific heat of 1.518 MJm<sup>-3</sup>K<sup>-1</sup> with an elastic modulus of one MPa

[8]. Comparable RTV elastomers have a glass transition temperature around 160 Kelvin
[9]. Combining RTV-655 with a material of lower density and lower thermal conductivity may improve the tank's thermal performance. The lower thermal conductivity will increase cryogenic liquid life and a lower density decreases the rocket's mass penalty.

Aerogels are porous solids formed by drying a gel solution without shrinking it by the process depicted in Figure 1 [10]. The resulting material has microscopic pore sizes (4-10 nm) giving aerogel its low thermal conductivity and density, putting them among the lightest known solid materials. Aerogels are ever increasing in popularity with proposed uses from biomedical to astronautical industries for their porosity and thermal resistance. The most common aerogel is native silica aerogel. Native silica aerogel demonstrates one of the lowest aerogel thermal conductivities, but it is too brittle for pressurized tank applications [11]. The properties of a silica aerogel can be tuned by manipulating the synthesis process as well as cross-linking another polymer to the base polymer. Cross-linking aerogel is useful in increasing the mechanical strength [12], while consequently increasing the thermal conductivity [11]. Also, aerogels can be synthesized using other types of polymers such as polyimide.



Figure 1: Process for synthesizing aerogels [10]

Polyimide aerogel is created by cross-linking polyimides in solution before gelling and drying the gel in a super critical point dryer, SCD. The properties of polyimide aerogel are dependent on the constituents in the synthesis process [13]. Polyimide aerogel was chosen because of its superior mechanical strength compared to native silica aerogel and polyurea cross-linked aerogel while maintaining a low thermal conductivity [11]. The polyimide aerogel used for measurements in this study was synthesized by NASA [13]. Another formulation of polyimide aerogel was synthesized by NASA using a unique mixture of constituents; the resulting polyimide aerogel produced a measured density around 100 kgm<sup>-3</sup> and a measured thermal conductivity of 14 mWm<sup>-1</sup>K<sup>-1</sup> [13]. However, the material properties of an aerogel will change based on several factors such as the general synthetization process and age of the aerogel. Thermal conductivity of the polyimide aerogel used in this study was previously measured with the TPS 1500 resulting in an average measured thermal conductivity of 0.398 Wm<sup>-1</sup>K<sup>-1</sup> [14].

It is hypothesized that the variation in thermal conductivity and volumetric specific heat of the RTV-655/polyimide aerogel compound material will be dependent on the ratio of the volume of polyimide aerogel to the total volume of the sample, known as volume ratio in this study. However, the combination of RTV-655 and polyimide aerogel proposed in this study creates a heterogeneous compound material. Many heterogeneous compound materials, like wood, fiber-reinforced material, and layered materials such as forms of MLI, have anisotropic properties, which vary directionally. Thus, the orientation of the embedded polyimide aerogel within the RTV-655 is carefully considered with respect to the inherent orientation of the experiment setup and method utilized to obtain the properties of interest and the layering used in the prototype cryogenic propellant tank. The relationship between volume ratio and thermal conductivity, as well as volumetric specific heat, is obtained by measuring different samples of the compound with varying

volume ratios. A better understanding of the relationship between the thermal properties and volume ratio is needed to assess the application of the RTV-655/polyimide aerogel compound material for future use in the construction of cryogenic liquid storage tanks.

#### <u>1.3 Thermal Characterization</u>

In general, thermal characterization encompasses determining the values for properties of a material that quantify how a material reacts to a heat fluctuation. In this study, the thermal characterization focuses on measuring the material properties related to heat transfer and more specifically the conduction mode of heat transfer. Thus, the material properties of interest in this study are thermal conductivity, thermal diffusivity, and volumetric specific heat. Characterizing conduction for varying volume ratios of the RTV-655/polyimide aerogel compound at a range of temperatures is important considering conduction through multi-layer insulation is the largest heat leak of a cryogenic storage system as discussed in Section 1.1. Convection in cryogenic liquid storage tanks is shown to diminish with reduction in gravity; convection outside the tank is non-existent due to the lack of fluid molecules, therefore, convection can be ignored. Radiation acts as the heat source in space which can be reduced using reflective wrapping. The present study focuses on characterizing the conduction heat transfer through the tank wall.

Thermal conductivity is a material property that quantifies the ability of a material to conduct heat in a certain direction. Thermal conductivity is derived as a proportionality constant in Fourier's law of heat conduction with SI units of Wm<sup>-1</sup>K<sup>-1</sup>, assuming a constant temperature difference. Cryogenic tanks in space experience thermal cycling, such that the conduction through the material is unsteady. Modeling unsteady heat

conduction through a material requires knowledge of thermal conductivity and volumetric specific heat, which create the combined material property of thermal diffusivity. Specific heat, normalized by mass, is the amount of energy required to increase an amount of mass of a material one degree in absolute scale with SI units of Jkg<sup>-1</sup>K<sup>-1</sup>. Volumetric specific heat, normalized by volume, is the product of specific heat MJm<sup>-3</sup>K<sup>-1</sup> and density kgm<sup>-3</sup> with SI units of MJm<sup>-3</sup>K<sup>-1</sup>. The ratio of thermal conductivity to volumetric specific heat is the thermal diffusivity of a material with SI units of mm<sup>2</sup>s<sup>-1</sup>. Physically, thermal diffusivity relates the ability of a material to conduct heat to the ability of a material to absorb heat.

The thermal conductivity, thermal diffusivity, and volumetric specific heat of a substance are a function of temperature requiring measurements at each temperature of interest. Also, the value of thermal conductivity and volumetric specific heat for the RTV-655/polyimide aerogel compound changes as the volume ratio changes. Thermal conductivity in a layered heterogeneous material will vary directionally. For this study, the polyimide aerogel layers are oriented to simulate the layering technique used in the prototype cryogenic propellant tank. If the total volume and cross-sectional area of the compound materials used in this study are equivalent as a means of experiment control, then changes in the thickness of polyimide aerogel in the compound will be the only significant variable length. A change in the ratio of the thicknesses is the only geometric parameter which can change the volume ratio for this study. The volume ratio is subsequently used as an independent variable for determining thermal conductivity with the understanding that the thickness ratios of the compound materials are only variable geometric parameters.

Specific heat is a property normalized by the mass of a material rather than the geometry of the material. However, volumetric specific heat is specific heat normalized by a dimension of length. As mentioned, if the total volume and cross-sectional area of the compound materials used in this study are equivalent as a means of experiment control, then changes in the thickness of polyimide aerogel in the compound will be the only variable length. A change in the ratio of the thickness is the only geometric parameter which can change the volume ratio for this study. The volume ratio is subsequently used as an independent variable for determining volumetric specific heat with the understanding that the thickness ratios of the compound materials are the only variable geometric parameter. Thermal conductivity, volumetric specific heat, and thermal diffusivity of RTV-655, polyimide aerogel, and three volume ratios of the compound were measured at 85K, 253K, 295K, and 313K using the hot disc transient plane source method in accordance with the ISO 22007-2:2015 standard [15].

#### <u>1.4 Previous Research</u>

This study presents thermal property measurements which are essential in the development of future computational simulations. The simulations can be subsequently used for modeling systems that can potentially benefit from the use of the RTV-655/polyimide aerogel compound material. A polymer and aerogel were proposed as a novel compound for constructing cryogenic liquid tanks for use in space. RTV-655 was chosen as the substrate due to its thermal properties and space-qualification. Polyimide aerogel was chosen following studies of the mechanical and thermal properties of other aerogels available at the time. Polyimide aerogel was the best option because of its ductility and availability in flexible thin sheets ideal for cylindrical tank designs. A small-

scale feasibility study was performed using two tanks: an RTV-655 tank as a control and an RTV-655/polyimide aerogel tank [11]. The tanks were filled with liquid nitrogen to observe the difference in thermal and mechanical performance of the two tank materials. The experimental results demonstrate a decrease in self-pressurization rates with the addition of aerogel and support the concept of using RTV-655 and polyimide aerogel to construct a cryogenic liquid container [11]. Computational models can be used to simulate large scale applications of the compound material. Accuracy of the computational model depends on accurate measurements of the material properties used in the computational model, which is the purpose of this study.

#### CHAPTER 2.

#### METHODS AND MATERIALS

#### 2.1 Transient Plane Source Method

Dr. Silas E. Gustafsson and his team in the Department of Physics at Chalmers University of Technology in Gothenburg, Sweden developed and patented the transient plane heat source method, TPS, as a rapid technique of measuring the thermal transport properties of solids, liquids, powders, and pastes from cryogenic to extremely high temperatures, 1000K [16]. The TPS method can determine thermal conductivity, thermal diffusivity, and volumetric specific heat of a material with a single measurement using a single sensor. The same machine can also be used to measure volumetric specific heat independently with two measurements: a reference measurement and a sample measurement. The machine that employs the TPS method for measuring both thermal conductivity and volumetric specific heat is known as a Hot Disk Thermal Constants Analyzer. The specifications and documented capabilities for six available Hot Disk Thermal Constants Analyzer models including the model used for this study, the TPS 1500, are listed in Table 1.

| TPS<br>Model | Minimum<br>Sample<br>Size | Thermal<br>Conductivity<br>Range    | Temperature<br>Range |
|--------------|---------------------------|-------------------------------------|----------------------|
|              | [mm]                      | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [K]                  |
| 3500         | 2                         | 0.005 to 1800                       | 113 to 1273          |
| 2500 S       | 2                         | 0.005 to 1000+                      | 113 to 1273          |
| 2200         | 6                         | 0.01 to 500                         | 113 to 1023          |
| 1500         | 13                        | 0.01 to 20+                         | 113 to 1023          |
| 500 S        | 13                        | 0.03 to 200                         | 113 to 573           |
| 500          | 6                         | 0.03 to 100                         | 113 to 573           |

Table 1: Hot Disk Thermal Constants Analyzer models and specifications.

The TPS 1500 Hot Disk Thermal Constants Analyzer, pictured in Figure 2, was purchased from ThermTest, Inc. As shown in Table 1: Hot Disk Thermal Constants Analyzer models and specifications., the TPS 1500 has the capability of measuring thermal conductivity and volumetric specific heat of materials within the temperature range of 113K to 1023K and between the thermal conductivity range of 0.01 and 20 Wm<sup>-1</sup>K<sup>-1</sup>. The machine uses thin, circular transient plane source sensors, known as hot disks, of varying radii based on the dimensions of the material being measured. The sensor, illustrated in Figure 3., is a thin, double nickel spiral covered by a thin protective layer of Kapton insulation. The sensor size and sensor number for the available hot disks are listed in Table 2.

| Radius |
|--------|
| [mm]   |
| 3.189  |
| 6.403  |
| 9.908  |
| 14.61  |
|        |

Table 2: Compatible hot disk sensor sizes and sensor numbers.



Figure 2: TPS 1500 Thermal constants analyzer.



Figure 3: Hot disk transient plane source sensor

### 2.1.1 Hot Disk Sensor Functions

The hot disk sensor, shown in Figure 3, has two functions: supply a constant heat flux to the sample and simultaneously measure the time-dependent temperature increase of the sensor's surface. The recorded transient temperature is the average temperature increase of the sensor's surface obtained by recording the transient resistance of the sensor. The

electrical resistivity of the nickel sensor changes with temperature and the rate of resistance change is a material property known as the thermal coefficient of electrical resistivity with SI units of (K<sup>-1</sup>), abbreviated as TCR in the software. Using the TCR value at the measurement temperature, the transient resistance of the sensor is denoted as:

$$R(t) = R_0 \left[ 1 + TCR \left( \Delta T_i + \Delta T(t) \right) \right] \tag{1}$$

Rearranging the equation for the transient temperature increase yields:

$$\Delta T(t) = \frac{1}{TCR} \left( \frac{R(t)}{R_0} - 1 \right) - \Delta T_i$$
(2)

Two-hundred evenly distributed points are recorded for all measurement times producing a graph like the one shown in Figure 4.



Figure 4: Transient temperature increase of hot disk sensor surface.

Also, before heat is supplied to the sample, the temperature of the sensor is measured for forty seconds. The difference between the initial temperature and the subsequent measurements is plotted on a graph called temperature drift. The temperature drift indicates if the sample was at steady state before heat was supplied to ensure no external heat sources are present during the measurement. A temperature drift graph indicating a steady state is shown in Figure 5.



Figure 5: Recorded temperature drift

The TCR value is a function of temperature and is available in the software between 113K and 1023K. The lack of TCR values below 113K require calibrating the TCR values at each temperature of interest below 113K. The TCR value at any temperature of interest can be calibrated by measuring a reference material with a measured reference thermal conductivity at the temperature of interest. The thermal conductivity of the reference material is measured at the experimental temperature with an appropriately sized hot disk sensor. The measured thermal conductivity from the software is equivocated to the measured reference value by adjusting the TCR value until the measured thermal conductivity matches the measured reference value. The same process is repeated for separate measurements. The optimal TCR value for each thermal conductivity measurement is recorded. The average TCR value from all the measurements becomes the newly calibrated TCR value at the respective measurement temperature. In this study, the calibrated TCR value is used for thermal conductivity and volumetric specific heat measurements.

As previously stated, a single measurement is enough to obtain the thermal conductivity, thermal diffusivity, and volumetric specific heat of a homogeneous material without knowing any of the material properties beforehand. However, the measurement of heterogeneous materials is known to provide larger uncertainties for measured thermal diffusivity and calculated volumetric specific heat. Consequently, volumetric specific heat was measured independently for RTV-655, polyimide aerogel, and three volume ratios of the RTV-655/polyimide aerogel compound at 85K, 253K, 295K, and 313K. The thermal conductivity and volumetric specific heat measurement methods involve two separate experimental setups.

#### 2.1.2 Thermal Conductivity Measurement Method

Thermal conductivity measurements begin with placing the sensor in between two identical, flat pieces of material large enough so that the sensor is at least the sensor's radius away from the outside of the material at all points illustrated in Figure 6.



Figure 6: Proper double-sided thermal conductivity measurement [17]

A piece of polystyrene foam can replace one of the pieces to perform a one-sided experiment, but for this study the sensor is placed between two nearly identical samples for double-sided measurements. Figure 6 shows the thermal conductivity sample holder used for measurements at 253K to 313K. After the sample and sensor have been secured, the measurement parameters such as time, temperature, power, mass, and minimum probing depth are input then the measurement is initiated. The raw measurement results appear after the measurement is conducted in the form of a temperature drift graph and a transient temperature graph. The temperature drift graph is used to determine if the sample was at steady state before the sample measurement was initiated. If the variation of temperature difference does not show any non-random fluctuation within the temperature drift graph, the sample is considered to have been at a steady state temperature before the measurement was conducted. The transient temperature graph contains the raw data used to facilitate the calculation of thermal conductivity and volumetric specific heat. A range of points is selected from the transient temperature graph to calculate the thermal properties. The software executes a linear optimization algorithm on an analytical model for the average temperature increase across the surface of the hot disk sensor to compute the thermal conductivity and thermal diffusivity, and then the volumetric specific heat can be solved for by using eq.  $\rho C = \frac{k}{\alpha}$  (8). Correspondingly, in this study, the solved volumetric specific heat is named the calculated volumetric specific heat and the result of the volumetric specific heat measurements is named the measured volumetric specific heat. The derivation of the TPS theory for measuring thermal conductivity and volumetric specific heat can be found in literature [18]. The analytical equation for the average temperature increase of the surface of any hot disk sensor is shown below in eq. (3), which is the equation that allows measuring thermal conductivity and thermal diffusivity simultaneously:

$$\bar{T}(\tau) = \frac{P_0}{\pi^{\frac{3}{2}} \cdot r \cdot k} D(\tau) + \Delta T_i$$
(3)

The temperature increase is proportional to  $D(\tau)$ , which is a geometric time dependent function defined as:

$$D(\tau) = \frac{1}{m^2(m+1)^2} \int_0^{\tau} \frac{d\sigma}{\sigma^2} \sum_{k=1}^m k \sum_{l=1}^m l e^{\frac{-\left(\frac{k^2+l^2}{m^2}\right)}{4\sigma^2}} I_0(\frac{kl}{2m^2\sigma^2})$$
(4)

The variable,  $\tau$ , is calculated and provided by the software as a variable named the total to characteristic time. The total to characteristic time is a dimensionless ratio of the elapsed measurement time within the measurement window to the characteristic time of the material being measured:

$$\tau = \sqrt{\frac{t}{\Theta}} \tag{5}$$

The characteristic time is an important aspect to the TPS theory because the characteristic time of the material normalizes the analytical model for any thermal diffusivity,  $\alpha$ , and size sensor, r, calculated as:

$$\Theta = \frac{r^2}{\alpha} \tag{6}$$

The software executes a computational routine to determine the line of best fit for the experimentally recorded temperature change versus  $D(\tau)$  by optimizing the thermal diffusivity value in the total to characteristic time,  $\tau$  [18]. The y-intercept of the line of best fit is used to determine the temperature gradient across the protective Kapton insulation of the hot disk sensor. The value for the slope of the line of best fit is set equal to the analytical slope defined in eq. (3) resulting in the following expression for thermal conductivity:

$$k = \frac{P_0}{\pi^{\frac{3}{2}} * r * slope} \tag{7}$$

The calculated volumetric specific heat can be solved dividing thermal conductivity by thermal diffusivity as shown in eq. (8) below:

$$\rho C = \frac{k}{\alpha} \tag{8}$$

The penetration of heat into the sample, known as the probing depth, is analytically found using the following relation:

$$p = \sqrt{4\alpha t} \tag{9}$$

It should be noted that the thermal conductivity and thermal diffusivity provided by the thermal conductivity measurements is the geometric mean of the radial and axial thermal conductivity and thermal diffusivity, known as the effective thermal conductivity, shown in eq. (10):

$$k = \sqrt{k_r \cdot k_a} \tag{10}$$

#### 2.1.3 Volumetric Specific Heat Measurement Method

The volumetric specific heat measurement employs the hot disk sensor differently than thermal conductivity measurements. The hot disk sensor is attached to the bottom of a copper cup, 4 cm in diameter and 11 mm in depth for this study, fitted with a lid and a rubber O-ring gasket around the top edge. The copper cup, lid, and sensor are enclosed in insulation to maximize the heat absorbed by the cup. The setup is secured by a sample holder designed for measurements in the ambient air, which was useful for the measurement temperatures of 253K, 295K, and 313K. The sample holder, insulation, and copper cup for the volumetric specific heat measurement are shown in Figure 7.



Figure 7: Volumetric specific heat measurement setup for 253K, 295K, and 313K.

Measuring volumetric specific heat includes two measurements: a reference measurement of the empty cup and a sample measurement of the cup containing the sample. The measurement process begins by supplying a certain amount of power for a specified amount of time to the empty sample holder in the reference measurement. After the reference measurement successfully conducted and saved, the reference measurement is used to initialize a sample measurement. Consequently, the reference measurement constrains the measurement time and temperature of the sample measurement. So, for every time and temperature, there must be at least one reference. The sample measurement calls for the mass and/or volume of the sample as well as an increase in the power input for the sample measurement to equivocate the sample and reference measurements. Following the sample measurement, the measured volumetric specific heat of the sample is obtained within from the software using points 100 to 200 for every material. The software provides specific heat and/or volumetric specific heat depending on whether mass and/or volume are input to the software. As mentioned in Section 1.3, specific heat is normalized by mass, whereas volumetric specific heat is normalized by volume. The fundamental calculation of specific heat requires the known power input for the sample and reference experiment, mass of the sample, reference measurement temperature difference, and time interval in the eq. (11) as follows:

$$C = \left(P_{sam} - P_{ref}\right) \frac{\Delta t}{M * \Delta T} \tag{11}$$

Measured specific heat can be normalized by a dimension of length when multiplied by the density of the material,  $\rho = \frac{M}{v}$ . The density of the material cancels out the mass term of specific heat and replaces it with volume shown in eq. (12) below. The software can also provide measured volumetric specific heat of a material knowing only the measured material volume shown in the last part of eq. (12)

$$\rho C = \frac{M}{V} \left( P_{sam} - P_{ref} \right) \frac{\Delta t}{M * \Delta T} = \left( P_{sam} - P_{ref} \right) \frac{\Delta t}{V * \Delta T}$$
(12)
Since the total volume of material is constrained by the volume of the copper cup, the mass of the material is the only changing variable. A variation in the compound sample mass within a fixed total volume can be reflected by the volume ratio of the sample, which is hypothesized to be an independent variable of the volumetric specific heat measurements. If the mass or density of a measured sample is considerably low because the volume ratio is high, the sensitivity of equivocating the reference temperature increase and sample temperature increase is exacerbated, which can lead to larger experiment uncertainty. The measured volumetric specific heat can be compared to the calculated volumetric specific heat obtained in thermal conductivity measurements. In general, eq.  $\rho C = \frac{M}{V} (P_{sam} - P_{ref}) \frac{\Delta t}{M*\Delta T} = (P_{sam} - P_{ref}) \frac{\Delta t}{V*\Delta T}$  (12(12) illustrates measured volumetric specific heat as the additional heat required to increase the temperature of the copper cup with the sample of a certain volume to the same amount as without the sample.

#### 2.2 Materials

Thermal conductivity and volumetric specific heat were measured for three groups of materials: reference, homogeneous, and heterogeneous. The reference materials were chosen to have a thermal conductivity within the measuring capabilities of the machine and documented thermal properties at the temperatures of interest. Paraffin wax, SS316, and XPS were chosen as the reference materials to validate and benchmark the measurement technique before measuring materials with unknown thermal properties. For this study, the homogeneous group of materials refers to RTV-655 and polyimide aerogel. The heterogeneous group of materials refers to the RTV-655/polyimide aerogel

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compound samples. RTV-655 and compound RTV-655/polyimide aerogel samples were made specifically for the thermal conductivity and volumetric specific heat measurement constraints. The samples used for thermal conductivity were molded in an aluminum block with a cylindrical pocket 13 mm deep and 38 mm in diameter. The 38 mm diameter was chosen to occupy the most surface area in the copper cup used for volumetric specific heat measurements. Additionally, a sample diameter of 38 mm allows a sensor of specific radius, sensor 5501 with a radius of 4.403 mm, to probe the entire available radial and axial probing depth. The sample thickness of 13 mm thickness is nearly twice the diameter of the sensor allowing the whole sample thickness to be probed while remaining within the recommended total time to characteristic time. Two samples were made for RTV-655 and three volume ratios of the RTV-655/polyimide aerogel compound to conduct double-sided thermal conductivity measurements. The volumetric specific heat measurements only required a single sample of RTV-655 and three volume ratios of the RTV-655/polyimide aerogel compound. The volumetric specific heat samples were molded in the same aluminum block with an 8 mm thick disc of the same diameter within the cylindrical hole to reduce the depth from 13 mm to 5 mm following the considerations for the volumetric specific heat measurement discussed in Section 2.3.2.

#### 2.2.1 Reference Materials

The supplier of the hot disk thermal constants analyzer, ThermTest, provided two stainless steel 316, SS316, discs along with a measured value at room temperature to validate the rated accuracy and precision of the TPS 1500. The SS316 discs, pictured in Figure 8, have a diameter of 6 cm and a thickness of 2 cm with a density of 8000 kgm<sup>-3</sup>. ThermTest provides a reference thermal conductivity for SS316 at room temperature of

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13.53 Wm<sup>-1</sup>K<sup>-1</sup>, which was measured by their own TPS system [19]. Also, thermal conductivity and volumetric specific heat of SS316 is available from 1K to 1700K within literature [20] [21] [22]. The thermal conductivity of SS316 at 295K is below the upper thermal conductivity limit of 20 Wm<sup>-1</sup>K<sup>-1</sup> for the TPS 1500. However, the thermal conductivity, specific heat, and density of SS316 is not akin to the thermal conductivity, specific heat, and density of SS316 is not akin to the thermal conductivity, specific heat, and density of interest, RTV-655 and polyimide aerogel. For this reason, reference materials with a lower thermal conductivity and density closer to that of RTV-655 and polyimide aerogel became desirable.



Figure 8: SS316 discs used as reference samples



Figure 9: Specific heat of SS316 as a function of temperature

Initially, paraffin wax was chosen as a benchmark material to replicate a documented volumetric specific heat measurement of paraffin wax at room temperature available from ThermTest [23]. Paraffin wax has a volumetric specific heat of 2.250  $MJm^{-3}K^{-1}$  and a thermal conductivity of 0.25  $Wm^{-1}K^{-1}$  at room temperature [24]. The benchmark thermal diffusivity of paraffin wax at 295K is 0.111 mm<sup>2</sup>s<sup>-1</sup>, which is almost identical to the benchmark thermal diffusivity for RTV-655 at 295 of 0.112 mm<sup>2</sup>s<sup>-1</sup>. Paraffin wax has a density of 900 kgm<sup>-3</sup> closer to the density of RTV-655, 1040 kgm<sup>-3</sup>, than to the density of polyimide aerogel, approximately 0.1 kgm<sup>-3</sup> [13]. Circular samples of paraffin wax, pictured in Figure 10, were shaped out of a square block to fit within the copper cup used in the volumetric specific heat measurements. The benchmark thermal conductivity measurements were conducted using the square paraffin wax samples. Reference values for the thermal conductivity and volumetric specific heat of paraffin wax are only available at room temperature [24] [23]. Consequently, the benchmark measurements for volumetric specific heat and thermal conductivity of paraffin wax was constrained to the measurement temperature of 295K.



Figure 10: Paraffin wax samples

Extruded polystyrene foam, XPS, is a rigid insulation formed in an extrusion process of creating tiny closed cells of a polystyrene polymer. As a result of the porosity, XPS has a low density and a low thermal conductivity useful for numerous insulation

applications. The XPS measured in this study, shown in Figure 11, was manufactured by Owen-Corning with measured thermal conductivities from ThermTest at 113, 273, and 293K in accordance with the ASTM C177 standard [25]. National Institute of Standards and Testing, NIST, provides thermal conductivity and volumetric specific heat for an XPS density of 32 kgm-3 [26]. Properties were also defined for a larger variation of density and thermal conductivity in [27] [28]. The reference thermal conductivity value of XPS at 85K was used to calibrate the TCR value of the hot disc sensor at 85K. The calibration process is explained in detail within Section 2.4. Thermal conductivity measurements were conducted using a pair of square foam samples with dimensions of 7 cm x 7 cm x 1 cm, pictured in Figure 11. The volumetric specific heat measurement method and experimental setup constrain the sample dimensions to a diameter of 38 mm or less and a thickness of 5 mm or less. The volume constraint creates a disproportionality between the sample mass of the high-density materials, RTV-655 and the three volume ratios, and the sample mass of the low-density materials, XPS and polyimide aerogel. Previous thermal conductivity measurements of RTV-655 and polyimide aerogel also used the polystyrene samples to calibrate the measurement software [14].



Figure 11: Extruded polystyrene blocks (left) and disc (right)



Figure 12: Thermal conductivity of XPS foam based on density and temperature.

# 2.2.2 Homogeneous Materials

As mentioned in Section 1.2, the polyimide aerogel measured in this study was synthesized by NASA [13]. The properties of the polyimide aerogel are known to change based on variations in the synthetization process as well as the constituents of the synthetization process [13]. The polyimide aerogel used in this study does not have measured reference property values at any temperature, meaning the thermal properties must be measured. The polyimide aerogel is available in two forms: monolith blocks with dimensions of 2 cm x 2 cm x 1 cm and thin, flexible sheets, which is the exact aerogel used to construct the prototype RTV-655/polyimide aerogel propellant tank. Consequently, the polyimide aerogel sheets were chosen as the desired form of polyimide aerogel to be measured in this study. Both forms of aerogel are pictured below in Figure 13.



Figure 13: Polyimide aerogel monolith block (left) and sheets (right).

RTV-655 is obtained as a two-part, part A and part B, compound that must be mixed, outgassed, and cured. The RTV-655 sample preparation begins by mixing part A and part B at a 10:1 mass ratio on a high precision scale. The amount of uncured RTV-655 used for making samples is small, as a result, the two parts should be mixed for 10 minutes. Mixing RTV-655 introduces a significant amount of air that needs to be outgassed before curing the RTV-655. For this study, RTV-655 was cured for one hour at a temperature of 373K, in accordance with the instructions provided by the manufacturer for complete curing [8]. A molded and cured RTV-655 disk is pictured in Figure 14. The thermal conductivity of another RTV silicone rubber, RTV-566, is available in literature from 1.2K to 300K shown in Figure 15 [29], which may be useful for comparing the thermal conductivity results of this study.



Figure 14: RTV-655 molded and cured into a disc.



Figure 15. Thermal conductivity of a different elastomer RTV-566 from 1.2K to 300K. 2.2.3 Heterogeneous Materials

The compound RTV-655/polyimide aerogel samples were made by embedding circular polyimide aerogel sheets within RTV-655 cured in the same 38 mm diameter aluminum mold. The circular polyimide aerogel sheets were cut with a 1-3/8 inch, nearly 35 mm, circular punch from large sheets of polyimide aerogel. The 3 mm difference between the diameter of the aerogel discs and diameter of the mold allowed the RTV-655 to cure around the embedded aerogel because RTV-655 and polyimide aerogel do not laminate to each other. To ensure that the aerogel discs stayed in the center of the mold, an aluminum bracket and threaded rod attachment, pictured in Figure 16, were designed to hold the polyimide aerogel sheets in place during outgassing and curing. The outgassing time for the volume ratio samples was considerably higher than for RTV-655 alone. The outgassing process and final cured sample are shown in Figure 17.



Figure 16: Sample mold and aerogel placement holder.

The number of aerogel discs chosen was dependent on the thickness of each disc as well as the desired volume ratio. Target volume ratios of 0.17, 0.34, and 0.51 were chosen to be consistent with a previous elasticity study of the RTV-655/polyimide aerogel compound [30], where volume ratio is defined as:

$$VR = \frac{V_{PI}}{V_{RTV} + V_{PI}} \approx \frac{t_{PI}}{t_{RTV} + t_{PI}}$$
(13)

The volume ratio of the samples will vary slightly from the ratio of the thicknesses because the polyimide aerogel is fully encapsulated, thus a 'layer' of polyimide aerogel includes a very small amount of RTV-655. Therefore, volume ratios presented herein are assumed to be approximately equal to the ratio of thicknesses. The volume of aerogel in each sample was calculated using the thickness and diameter for each set of aerogel discs within a sample. The average measured mass, volume, polyimide aerogel density, and measured volume ratio for each sample set used in the thermal conductivity measurements is listed in Table 3. The average measured mass, volume, aerogel density, and measured volume ratio for each sample set used in the volumetric specific heat measurements is listed in Table 4. shows the progression of outgassing and a final cured compound RTV-655/polyimide aerogel sample. The cross-sectional view of the compound samples at each volume ratio was examined by a stereo microscope and images acquired during this examination are presented in Figure 18 for the thermal conductivity samples and in Figure 19 for the volumetric specific heat samples.

| Parameter    |                     | <b>VR20</b> | VR34     | VR56     |  |  |
|--------------|---------------------|-------------|----------|----------|--|--|
| Diameter     | [m]                 | 0.038       | 0.038    | 0.038    |  |  |
| Thickness    | [m]                 | 0.012       | 0.013    | 0.013    |  |  |
| Mass         | [kg]                | 0.013       | 0.012    | 0.011    |  |  |
| Volume       | [m <sup>3</sup> ]   | 1.32E-05    | 1.43E-05 | 1.52E-05 |  |  |
| Density      | [kgm <sup>3</sup> ] | 954         | 824      | 736      |  |  |
| PI Volume    | [m <sup>3</sup> ]   | 2.67E-06    | 4.86E-06 | 8.49E-06 |  |  |
| Volume Ratio |                     | 0.20        | 0.34     | 0.56     |  |  |

Table 3: Average dimensions for each volume ratio sample set used for the thermal conductivity measurements

Table 4: Average dimensions for each volume ratio sample set used for the volumetric specific heat measurements

| Parameter    |                     | VR20     | <b>VR47</b> | VR71     |
|--------------|---------------------|----------|-------------|----------|
| Diameter     | [m]                 | 0.038    | 0.038       | 0.038    |
| Thickness    | [m]                 | 0.005    | 0.006       | 0.006    |
| Mass         | [kg]                | 0.006    | 0.005       | 0.004    |
| Volume       | [m <sup>3</sup> ]   | 6.08E-06 | 6.23E-06    | 6.28E-06 |
| Density      | [kgm <sup>3</sup> ] | 939      | 808         | 698      |
| PI Volume    | [m <sup>3</sup> ]   | 1.19E-06 | 2.95E-06    | 4.46E-06 |
| Volume Ratio |                     | 0.20     | 0.47        | 0.71     |



Figure 17. RTV-655/ Polyimide sample preparation progress.







Figure 18. Cross-sectional view of VR20, VR34, and VR56 (left to right) used in thermal conductivity measurements.



Figure 19. Cross-sectional view of VR20, VR47, and VR71 (left to right) used in volumetric specific heat measurements.

#### 2.3 Methods

#### 2.3.1 Thermal Conductivity Measurement Considerations

Thermal conductivity measurements should include the following considerations. The total recorded temperature increase should be within 2K to 7K and the sample should be at a steady-state temperature before heat is supplied, which can be verified by inspecting the temperature drift. The penetration depth of the heat must be less than the difference between the radius of the cylindrical sample and radius of the sensor as well as the axial length of the cylindrical sample. The probing depth is determined using the solved thermal diffusivity and measurement time within eq.  $p = \sqrt{4\alpha t}$ (9). The total to characteristic time,  $\tau^2$ , should be between 0.333 to 1.0, which corresponds to a penetration depth of a radius and twice the radius of the sensor, respectively. Therefore, the best results require the sample to have a diameter greater than or equal to two times the diameter of the sensor and a thickness greater than or equal to the radius of the sensor. Four sizes of sensors were used within this study with the numbers and diameters listed in Table 2. The larger sensors, with a radius of 6.403 mm or above, measure heterogeneous materials more accurately due to the increased amount of material represented in the measured results [31]. The best calculations do not include the beginning points to neglect the effect of thermal contact resistance between the sensor and material interface. Once the properties are calculated, the mean deviation should be in the order of e-4.

The sensor should be firmly clamped, aligned between the samples, and taut as shown in Figure 6. The measurements at 253K, 295K, and 313K used the same sample holder as shown in Figure 6, while measurements at 85K require a separate sample

holder, pictured in



Figure 21. The recommended measurement time, power, and sensor is provided for each temperature under the respective heading below. If alternate sensors are used for measurements, the amount of time and power will need to be changed to satisfy the temperature and probing depth conditions for the new measurements.

# 2.3.1.1 Thermal Conductivity 313 Kelvin Method

Thermal conductivity measurements at 313K were performed inside of a Tenney jr. environmental chamber. The environmental chamber was able to minimize temperature drift maintaining the temperature at 313K plus or minus a tenth of a degree. Three sizes of sensors were used depending on the material being measured and the available probing depth for the samples being measured. SS316 and XPS were measured with the 8563 sensor because the available probing depth for both materials exceeded its radius of 9.908 mm. RTV-655 and the three volume ratios were measured the 5501 sensor, with a radius of 6.403 mm, to achieve similar probing depths in addition to the maximum probing depth constraint. The polyimide aerogel sheets were measured with the 5465 sensor, with a radius of 3.189 mm, due to the available probing depth of the stacked sheets. For each set of measurements, the samples were allowed at least five hours to reach a steady temperature of 313K before performing a set of five to ten measurements with thirty to sixty minutes in between each subsequent measurement. The prescribed time, power, and sensor for the thermal conductivity measurements at 313K are listed for each material in Table 5.

| Material        | Time | Power | Sensor |
|-----------------|------|-------|--------|
|                 | [s]  | [mW]  |        |
| VR0.0 (RTV-655) | 320  | 50    | 5501   |
| VR20            | 640  | 35    | 5501   |
| VR34            | 320  | 35    | 5501   |
| VR56            | 320  | 25    | 5501   |
| VR1.0 (PI)      | 80   | 7     | 5465   |
| SS 316          | 20   | 3000  | 8563   |
| XPS             | 80   | 24    | 8563   |

Table 5: Inputs for thermal conductivity measurements at 313K

#### 2.3.1.2 Thermal Conductivity 295 Kelvin Method

Thermal conductivity measurements at 295K were performed in the ambient conditions of the laboratory over the environmental chamber due to the instability of the chamber's control system at 295K. Three sizes of sensors were used for the measurements at 295K to maximize probing depth for each material. Stainless steel was measured with the 8563 sensor; XPS, RTV-655, and the three volume ratios were measured with the sensor 5501 to obtain similar probing depths; and polyimide sheets are restricted to the small 5465 sensor due to the available probing depth. After the sensor and sample have been properly secured in the correct sample holder, the samples are given three hours to obtain a steady state temperature before beginning measurements. At least five measurements are performed for each sample and time with thirty to sixty minutes in between depending on the thermal diffusivity of the material. The time in between measurements should be increased if a temperature drift is observed. Table 6 lists the time, power, and sensor used to measure each property at 295K.

|                 |       | 5       |        |
|-----------------|-------|---------|--------|
| Material        | Time  | Power   | Sensor |
|                 | [s]   | [mW]    |        |
| VR0.0 (RTV-655) | 320   | 40      | 5501   |
| VR20            | 640   | 25      | 5501   |
| VR34            | 640   | 20      | 5501   |
| VR56            | 320   | 20      | 5501   |
| VR1.0 (PI)      | 40-80 | 10 or 7 | 3563   |
| SS 316          | 20    | 3500    | 8563   |
| Paraffin Wax    | 160   | 100     | 5501   |
| XPS             | 40    | 8       | 5501   |

Table 6: Inputs for thermal conductivity measurements at 295K.

## 2.3.1.3 Thermal Conductivity 253 Kelvin Method

Thermal conductivity measurements at 253K were executed in a chest freezer. The temperature was recorded to be 253K plus or minus two degrees. The measurements in the freezer are susceptible to temperature drifts due to the cycling of the refrigeration system. This can be can be mitigated by enabling temperature drift compensation within the calculation window. Stainless steel 316, XPS, RTV-655, and the smallest volume ratio were measured using the 5501 sensor. A failure of the 5501 sensor forced the two larger volume ratios to be measured with the 8563 sensor, which is an acceptable option. The polyimide sheets require the 5465 sensor to accommodate its available probing depth. The time, power, and sensor used for each measured material is listed in Table 7.

| Material        | Time | Power | Sensor |
|-----------------|------|-------|--------|
|                 | [s]  | [mW]  |        |
| VR0.0 (RTV-655) | 320  | 50    | 5501   |
| VR20            | 640  | 30    | 5501   |
| VR34            | 320  | 70    | 8563   |
| VR56            | 320  | 20    | 8563   |
| VR1.0 (PI)      | 40   | 5     | 3563   |
| SS 316          | 20   | 1000  | 5501   |
| XPS             | 40   | 10    | 5501   |

Table 7: Inputs for measuring thermal conductivity at 253K.

# 2.3.1.4 Thermal Conductivity 85 Kelvin Method

Thermal conductivity measurements at 85K utilized a low temperature apparatus submerged in a dewar filled with liquid nitrogen, described in detail in a following section. The temperature was measured using a type K thermocouple, which is capable of measuring temperature at 85K. The time, power, and sensor are indicated for each measurement in Table 8. There was a significant decrease in power for the lower temperature measurements relative to the higher temperature measurements indicating thermal conductivity decreases with temperature. All the experiments required a longer time of at least 6 hours before measurements were initiated with at least ninety minutes in between to allow the sample to achieve steady state.

| Material        | Time        | Power    | Sensor |
|-----------------|-------------|----------|--------|
|                 | [s]         | [mW]     |        |
| VR0.0 (RTV-655) | 80 to 160   | 50 to 40 | 5501   |
| VR20            | 320 to 640  | 12 to 8  | 5501   |
| VR34            | 640         | 18       | 5501   |
| VR56            | 640 to 1280 | 20 to 16 | 5501   |
| VR1.0 (PI)      | 80 or 160   | 4 or 7   | 5465   |
| SS 316          | 20          | 750      | 8563   |
| XPS             | 20          | 5        | 5501   |

Table 8: Inputs for measurement of thermal conductivity at 85K.

# 2.3.2 Volumetric Specific Heat Measurement Considerations

Accurate volumetric specific heat measurements using the hot disk transient plane source method include the following considerations. The measurement times chosen should be greater than the thermal settling times of each material, listed in Table 9. The thickness of the sample and its thermal diffusivity are used in the following equation to determine thermal settling time:

$$T_{settling} = \sqrt{\frac{L^2}{\alpha}}$$
(14)

| dole 71 Estimated therm | uole 9. Estimated thermal setting time for an materials at each temperate |      |      |     |  |  |
|-------------------------|---|------|------|-----|--|--|
| Material                | 313K  | 295K | 253K | 85K |  |  |
|                         | [s]   | [s]  | [s]  | [s] |  |  |
| VR0.0 (RTV-655)         | 241   | 231  | 214  | 65  |  |  |
| VR20                    | 377   | 313  | 802  | 409 |  |  |
| VR34                    | 324   | 321  | 300  | 402 |  |  |
| VR56                    | 183   | 193  | 238  | 177 |  |  |
| VR1.0 (PI)              | 76  | 85   | 80   | 374 |  |  |
| XPS                     | 33  | 36   | 39   | 29  |  |  |

Table 9: Estimated thermal settling time for all materials at each temperature

It should be noted that the thermal diffusivity used for each calculation was from thermal conductivity measurements, which are known to include errors for the heterogeneous materials. The sample should be five millimeters or less in thickness to prevent occupying more than half the volume of the cup. The total temperature increase of the reference experiment for measured materials should be greater than or equal to 3K and less than or equal to 7K [25]. The sample holder and sensor should be well insulated to maximize the amount of supplied heat absorbed by the sample being measured. The ambient conditions between reference and sample measurements should be identical. The temperature for all measurements was monitored periodically using a type K thermocouple. An increase in power for the sample measurement is required to match the temperature increase of the reference measurement. An estimation for the required sample measurement power can be solved and calculated from eq.  $\rho C = \frac{M}{V} (P_{sam} - V)^{2}$ 

$$P_{ref}$$
) $\frac{\Delta t}{M*\Delta T} = (P_{sam} - P_{ref})\frac{\Delta t}{V*\Delta T}$ (12) using an educated guess for volumetric specific  
heat along with all known variables of the volumetric specific heat measurement. The  
sample, sensor, and holder should be at a steady state temperature which can be verified  
by inspecting the temperature drift graph. The temperature drift graph should be  
inspected for temperature drift to verify the sample holder is at a steady state. Points 100  
to 200 are used for the calculation for all measurements.

#### 2.3.2.1 Volumetric Specific Heat 313 Kelvin Method

Volumetric specific heat measurements were performed in an environmental chamber with a steady temperature of 313K plus or minus a tenth of a degree. The measurements were conducted in the environmental chamber to eliminate differences

between reference and sample measurement environments. The reference and sample power used for 313K measurements are recorded in Table 10.

| Material           | Mass  | Time | Reference<br>Power | Sample<br>Power |
|--------------------|-------|------|--------------------|-----------------|
|                    | [g]   | [s]  | [mW]               | [mW]            |
| VD0.0(DTV.655)     | 4 702 | 160  | 377                | 610             |
| V K0.0 (K I V-033) | 4.795 | 320  | 200                | 300             |
| VD20               | 5 709 | 160  | 377                | 605 to 615      |
| V K20              | 5.708 | 320  | 200                | 322             |
| VR47               | 5.038 | 160  | 377                | 575             |
|                    |       | 320  | 200                | 310             |
| VD71               | 1 200 | 160  | 377                | 560             |
| V K / 1            | 4.300 | 320  | 200                | 305             |
|                    |       | 80   | 660                | 608 to 705      |
| VR1.0 (PI)         | 0.512 | 160  | 377                | 378 to 400      |
|                    |       | 320  | 216                | 216.5 to 228    |

Table 10: Inputs for volumetric specific heat measurements of the compound material at 313K.

## 2.3.2.2 Volumetric Specific Heat 295 Kelvin Method

Volumetric specific heat capacity measurements were performed in the ambient air of the laboratory which has an average air temperature of 295K with a few degrees of fluctuation throughout the day. The ambient temperature of the laboratory experienced higher variability than within the environmental chamber at 313K discussed in Section 2.3.1.1. The room temperature measurements were performed for a large measurement time range and the inputs for each reference and sample measurement are recorded in Table 11.

| Material   | Mass    | Time | Reference<br>Power | Sample Power        |
|--|---------|------|--------------------|---------------------|
|  | [g]     | [s]  | [mW]               | [mW]                |
|  |         | 160  | 200                | 345 to 367.6        |
| $\mathbf{V}\mathbf{D}0 (\mathbf{D}\mathbf{T}\mathbf{V} 655)$ | 5 292   | 320  | 125                | 204 to 212          |
| V KU.U (KI V-033)  | 5.565   | 640  | 95                 | 140                 |
|  |         | 1280 | 80                 | 105                 |
|  |         | 160  | 200 to 377         | 400 to 700          |
| VR20   | 5 704   | 320  | 125 to 222         | 245 to 382          |
| VIX20  | 5.704   | 640  | 95 to 145          | 160 to 226          |
|  |         | 1280 | 80 to 107          | 100 to 147          |
|  |         | 160  | 200                | 375 to 385          |
| VR/17  | 5.038   | 320  | 125                | 227 to 235          |
| VIXT/  | 5.050   | 640  | 95                 | 153 to 158          |
|  |         | 1280 | 80                 | 95 to 98            |
|  | 4.388   | 160  | 200 to 377         | 385 to 580          |
| VR71   |         | 320  | 125 to 222         | 235 to 360          |
| V IX / 1   |         | 640  | 95 to 145          | 158 to 200          |
|  |         | 1280 | 80 to 107          | 98 to 140           |
|  | 1.610   | 160  | 234                | 278                 |
| VP10(PI)   |         | 320  | 141                | 106                 |
| VK1.0 (F1)   |         | 640  | 95                 | 162.8 to 163.1      |
|  |         | 1280 | 73                 | 79                  |
|  |         | 320  | 141                | 375                 |
| Paraffin Wax   | 8.735   | 640  | 95                 | 217 to 217.5        |
|  |         | 1280 | 73                 | 136                 |
|  |         | 20   | 1600               | 1640                |
|  |         | 40   | 1100               | 1121                |
|  |         | 80   | 550 to 600         | 558.62 to<br>669.87 |
| XPS  | 0 1 1 0 | 160  | 377                | 381 to 382.25       |
|  | 0.110   | 320  | 222                | 224.15 to 224.61    |
|  |         | 640  | 145                | 146.2 to 146.4      |
|  |         | 1280 | 107                | 107.62 to<br>107.63 |

Table 11: Inputs for volumetric specific heat measurements at 295K.

#### 2.3.2.3 Volumetric Specific Heat 253 Kelvin Method

Volumetric specific heat measurements at 253K were conducted in a chest freezer. The temperature was measured to be an average of 253K with fluctuations in temperature based on the refrigeration cycle. The inputs for all measurements at 295K are listed in Table 12.

| Material        | Mass  | Time | Reference Power | Sample Power |
|-----------------|-------|------|-----------------|--------------|
|                 | [g]   | [S]  | [mW]            | [mW]         |
|                 |       | 160  | 350             | 368          |
| VR0.0 (RTV-655) | 5.383 | 320  | 200             | 204          |
|                 |       | 640  | 135             | 140          |
|                 |       | 160  | 350             | 565 to 600   |
| VR20            | 5.704 | 320  | 200             | 315 to 335   |
|                 |       | 640  | 135             | 190 to 200   |
|                 | 5.038 | 160  | 350             | 550          |
| VR47            |       | 320  | 200             | 320          |
|                 |       | 640  | 135             | 185          |
|                 |       | 160  | 350             | 530          |
| VR71            | 4.388 | 320  | 200             | 310 to 320   |
|                 |       | 640  | 135             | 185 to 200   |
|                 | 0.512 | 160  | 350             | 360 to 373   |
| VK1.0 (P1)      | 0.512 | 320  | 200             | 210 to 225   |

Table 12: Inputs for volumetric specific heat measurements at 253K.

#### 2.3.2.4 Volumetric Specific Heat 85 Kelvin Method

Volumetric specific heat was measured at 85K using the setup and calibration method described in the low temperature apparatus section below. The TCR value from the thermal conductivity measurements was used for the volumetric specific heat measurement. The amount of power used for each measurement time was much lower than the higher temperature measurements because of the decrease in thermal conductivity of the materials in addition to an increase in the TCR value. The prescribed variables for each material measured at 85K is listed in Table 13. RTV-655, polyimide aerogel, and the largest volume ratio were measured the most because of the small changes in properties with the lower volume ratios.

| Material           | Mass           | Time | Reference<br>Power | Sample<br>Power |
|--------------------|----------------|------|--------------------|-----------------|
|                    | [g]            | [s]  | [mW]               | [mW]            |
|                    |                | 40   | 80                 | 165             |
| VD0.0(DTV.655)     | 15 002         | 80   | 80                 | 120             |
| V KU.U (K I V-033) | 13.992         | 160  | 60                 | 105 to 135      |
|                    |                | 320  | 40                 | 90              |
| VR20               | 13.032         | 160  | 60                 | 105             |
| VP47               | 11.920         | 160  | 60                 | 98 to 105       |
| V IX4 /            |                | 320  | 40                 | 65 to 75        |
|                    | 4.686          | 160  | 60 to 80           | 95 to 180       |
| VR71               |                | 320  | 40 to 70           | 65 to 150       |
|                    |                | 640  | 40 to 60           | 62.5 to 135     |
|                    |                |      |                    |                 |
| VR1.0 (PI)         |                | 40   | 120                | 126             |
|                    | 0.755 to 1.012 | 80   | 100                | 115             |
|                    | 0.755 10 1.012 | 160  | 80                 | 85 to 107       |
|                    |                | 320  | 60                 | 65 to 135       |
|                    |                | 640  | 40                 | 44              |

Table 13: Inputs for volumetric specific heat mesasurements at 85K.

# 2.4 Low Temperature Setup and Calibration

A previously constructed low temperature apparatus was repurposed for this study. The apparatus secures and isolates the sample and TPS sensor while they are immersed in liquid nitrogen within a 35-liter dewar. The apparatus consists of two components made of stainless steel: a sample and sensor holder and an enclosure to separate the sample and sensor from outside environment. The original holder required cutting the sensor cable and attaching each end of wires to either side of four electrically insulated threaded rods that pass through the holder top. Instead, a new, similar holder was designed to prevent damaging other sensors and allow using different sensors; the new design was fabricated out of stainless steel with a hole in the top letting the sensor cable pass through. The difference between the old and new sample holder component can be seen in Figure 20



Figure 20. Old (left and middle) and new (right) low temperature sample holders.

The redesigned low temperature sample holder fits within the same stainless-steel container as the old low temperature sample holder. Two metal rods are welded to two separate screws that are meant to be fasted on opposite sides for lowering and raising the setup out of the dewar, which are partially shown in the far-right picture of Figure 20. The opening of the dewar is typically filled by an insulation top; however, the insulation top cannot fit in the opening during measurements due to the sensor wire. So, the opening is occupied by a towel wrapped around the sensor cable to reduce the rate of liquid nitrogen vaporization. Figure 21 shows the low temperature apparatus and the final measurement setup within the dewar.

In the absence of published TCR values at temperatures below 113K, the thermal conductivity and volumetric specific heat measurements at 85K required the calibration the TPS 1500. The TCR value can be calibrated at any temperature using a reference material with a reference thermal conductivity at any temperature of interest. The reference material for calibrating the TCR value has a reference thermal conductivity of 10.1 mWm<sup>-1</sup>K<sup>-1</sup> at 85K, which is on the cusp of the lower thermal conductivity range for the TPS 1500. The measured thermal conductivity of XPS at 85K is calculated iteratively for different TCR values until the measured and reference thermal conductivity of XPS at 85K are as close as experimentally possible. ThermTest provided XPS samples to calibrate the TCR value at 85K. The sample environment was measured to be 85K with a type K thermocouple, slightly above the boiling point of liquid nitrogen, 77K, due to the temperature increase across the stainless-steel wall of the sample holder. The TCR value was calibrated to a value of 0.014373 K<sup>-1</sup> at 85K.



Figure 21. Cryogenic temperature apparatus and measurement setup.

# CHAPTER 3.

## RESULTS

#### 3.1 Validation Results

## 3.1.1 Validation of the TPS 1500 Accuracy and Precision

The accuracy and precision of the TPS 1500 was validated by comparing the measured thermal conductivities of the reference materials,SS316 and XPS, at 295K to reference thermal conductivities of SS316 and XPS at 295K provided by the manufacturer, ThermTest [19]. The thermal conductivities for the reference materials lie within the manufacturer's specified thermal conductivity range of 0.01 Wm<sup>-1</sup>K<sup>-1</sup> to 20 Wm<sup>-1</sup>K<sup>-1</sup> for the TPS 1500. ThermTest states that within the temperature range -160°C to 1000°C, results should have a percent error of less than 5% and a repeatability of less than 2% [32]. The average thermal property measured or calculated in each set of measurements is the value reported in this study. The uncertainty is determined by the sample standard deviation of the measurement set calculated for thermal conductivity, *k*, in the following example:

$$s = \sqrt{\frac{\sum (k - \bar{k})^2}{n - 1}} \tag{15}$$

The precision of the validation measurements is calculated as the relative sample standard deviation, *RSD*, of the measured property. An example of the relative standard deviation calculation for thermal conductivity is shown eq.  $RSD = \frac{s}{k} * 100$  (16) as follows:

$$RSD = \frac{s}{k} * 100 \tag{16}$$

The relative absolute error is calculated as the difference between the average measured property and reference value of a material, which is expressed as a percentage of the reference value as shown for average measured thermal conductivity,  $\bar{k}$ , in eq. e =

$$\frac{|\bar{k}-k_{ref}|}{k_{ref}} * 100$$
 (17):

$$e = \frac{|\bar{k} - k_{ref}|}{k_{ref}} * 100$$
(17)

The purpose of measuring reference materials is to ensure the functionality of the hardware and, more importantly, gain confidence in the measurement technique by comparing personally measured values to reference values.

The validation results for SS 316 and XPS at 295K are listed in Table 14. The average measured thermal conductivity of SS316 at 295K is 13.48 Wm<sup>-1</sup>K<sup>-1</sup> with a repeatability of 0.2%. The relative absolute error between the average measured thermal conductivity and reference thermal conductivity of SS316 at 295K is 0.4%. The average measured thermal conductivity of XPS at 295K is 0.029 Wm<sup>-1</sup>K<sup>-1</sup> with a standard deviation below the precision of the TPS 1500. The relative absolute error between the average measured thermal conductivity and reference thermal conductivity of XPS at 295K is 0.6%. The relative absolute error and repeatability for the validation measurements of SS316 and XPS at 295K are below the manufacturer's documented accuracy and precision capabilities, which validates the rated accuracy and precision of

the TPS 1500. Thus, all subsequent measurements within the manufacturer's specified temperature range, thermal conductivity range, and sample size range are assumed to be within the accuracy and precision ratings of the TPS-1500.

| Material | Number of<br>Measurements | Measured k                          | k<br>RSD | Reference<br>k                      | Relative<br>Absolute<br>Error |
|----------|---------------------------|-------------------------------------|----------|-------------------------------------|-------------------------------|
|          | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]                           |
| SS 316   | 10                        | $13.482 \pm 0.029$                  | 0.2%     | 13.530                              | 0.4%                          |
| XPS      | 10                        | $0.029 \pm 0.000$                   | 0.0%     | 0.029                               | 0.6%                          |

Table 14: Validation results for TPS method using SS 316 and XPS at295K

#### 3.1.2 Benchmark Material Thermal Property Measurement Results

Thermal conductivity and volumetric specific heat measurements of SS316, XPS, paraffin wax, and RTV-655 were benchmarked by comparing the measured properties to reference properties available in the literature. Note that the samples and reference values at 295K reported in Section 3.1.1. are provided by ThermTest to verify the accuracy and precision of the instrument at room temperature [19], while the property values used in the following benchmark measurements were obtained from a variety of sources. The goal of the benchmarking study is to gain confidence in the various experiment setups and methods over a range of temperatures and to compare thermal property measurements for materials that are expected to be representative of the RTV-655 and polyimide aerogel. The selection of reference materials for both the validation measurements and benchmark measurements is outlined in Section 2.2.1.

Although SS316 is not representative of either RTV-655 or polyimide aerogel, SS316 was chosen first because the samples used for the TPS 1500 accuracy and

precision validation study were available. More importantly, benchmark values for thermal conductivity and volumetric specific heat of SS316 at 85K, 253K, 295K, and 313K are available in the literature [20] [21] [22]. Paraffin wax was selected as a benchmark material for the volumetric specific heat measurements because a reference value is provided by ThermTest at room temperature. Paraffin wax was selected as a benchmark material for thermal conductivity measurements because the thermal conductivity of paraffin wax at room temperature is close in magnitude to the thermal conductivity of RTV-655 at room temperature. XPS was selected as a representative porous material with a low thermal conductivity, similar in magnitude to thermal conductivities of some aerogels. A reference value for the thermal conductivity of RTV-655, a material of interest for this study, is reported by the manufacturer at room temperature. The relative absolute difference between the measured and benchmark thermal property value is determined with eq.  $e = \frac{|\bar{k} - k_{ref}|}{k_{ref}} * 100$  (17), which was previously used to calculate the accuracy of the validation results. The resulting percentage differences reported in the benchmarking study are not to be considered solely with respect to the 5% accuracy of the measurement device because the singular benchmark values reported are typically for a sample study. Unlike SS316, additional margins of difference are expected for XPS, paraffin wax, and RTV-655 since the properties for these materials can vary more from one sample to another due to subtle differences in preparation and/or porosity.

The thermal conductivity of SS316 was measured and compared to the benchmark thermal conductivities of SS316 available at 313K, 295K, 253K, and 85K [21]. The results, recorded in Table 15, demonstrate SS316 thermal conductivity

consistently decreases as temperature decreases. The average percent difference between measured and documented thermal conductivity at 313K, 295K, 253K, and 85K are 3.0%, 1.0%, 2.9%, and 6.3%, respectively. Discussions with the manufacturer indicate that the instrument should still be able to reliably measure thermal conductivity outside of the recommended temperature range, however, the average measurement error may exceed the 5% accuracy rating. The 6.3% relative absolute difference between the measured and benchmark thermal conductivity of SS316 at 85K supports the manufacturer's conclusion based on published data for SS316. The average thermal conductivity of SS316 and reference values are plotted for each temperature in Figure 22.

| Temperature | Number of<br>Measurements | Average<br>Measured <i>k</i>        | k<br>RSD | Benchmark<br>k                      | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|-------------------------------------|----------|-------------------------------------|------------------------------------|
| [K]         | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]                                |
| 313         | 14                        | $14.101 \pm 0.055$                  | 0.4%     | 13.67                               | 3.2%                               |
| 295         | 10                        | $13.482\pm0.029$                    | 0.2%     | 13.35                               | 1.0%                               |
| 253         | 5                         | $12.929 \pm 0.018$                  | 0.1%     | 12.57                               | 2.9%                               |
| 85          | 10                        | $8.317 \pm 0.025$                   | 0.3%     | 7.83                                | 6.3%                               |

Table 15: Thermal conductivity benchmark measurement results for Stainless Steel 316



Figure 22: Temperature dependence of Stainless Steel 316 thermal conductivity.

As described in Section 2.1.2., thermal conductivity measurements also provide a measured thermal diffusivity and a calculated volumetric specific heat. Thermal conductivity results for measured thermal diffusivity and calculated volumetric specific heat suffice for homogeneous materials because the calculation assumes the sample has constant density throughout. Since the TPS method is inherently directional when a sample is probed, ThermTest recommends measuring volumetric specific heat exclusively for heterogeneous materials rather than relying on the calculated volumetric specific heat. Discerning the behavior of all the measurement results for homogeneous materials is important before measuring heterogeneous samples of the combination of RTV-655 and polyimide aerogel at varying volume ratios. The average measured thermal diffusivity and relative standard deviation, *RSD*, of the SS316 measurements are listed in Table 16. Thermal diffusivity of SS316 decreases from 313K to 295K followed by a constant increase until its maximum value of 4.74 mm<sup>2</sup>s<sup>-1</sup> at 85K. The RSD is higher for thermal diffusivity than for thermal conductivity of SS316 at each temperature. The

higher RSD reported for the thermal diffusivity measurements when compared to the thermal conductivity measurements confirms the TPS method results in less uncertainty in measuring thermal conductivity of a material.

| Temperature | Number of<br>Measurements | Average Measured $\alpha$          | α RSD |
|-------------|---------------------------|------------------------------------|-------|
| [K]         | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]   |
| 313         | 14                        | $3.594 \pm 0.026$                  | 0.7%  |
| 295         | 10                        | $3.060\pm0.007$                    | 0.2%  |
| 253         | 5                         | $3.565\pm0.042$                    | 1.2%  |
| 85          | 10                        | $4.744 \pm 0.067$                  | 1.4%  |

Table 16: Thermal diffusivity benchmark measurement results for Stainless Steel 316

The volumetric specific heat of SS316 is calculated within the software by dividing the measured thermal conductivity by the measured thermal diffusivity for each temperature. The calculated volumetric specific heat,  $\rho C$ , is provided for SS316 in Table 17. The calculated volumetric specific heat of stainless steel 316 increases from 3.923 MJm<sup>-3</sup>K<sup>-1</sup> at 313K to a value of 4.405 MJm<sup>-3</sup>K<sup>-1</sup> at 295K followed by a decrease in value until the calculated volumetric specific heat of 1.753 MJm<sup>-3</sup>K<sup>-1</sup> at 85K.

| Temperature | Number of<br>Measurements | Average calculated $\rho C$          | ρC<br>RSD |
|-------------|---------------------------|--------------------------------------|-----------|
| [K]         | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]       |
| 313         | 14                        | $3.923 \pm 0.016$                    | 0.4%      |
| 295         | 10                        | $4.405 \pm 0.007$                    | 0.2%      |
| 253         | 5                         | $3.627 \pm 0.048$                    | 1.3%      |
| 85          | 10                        | $1.753 \pm 0.022$                    | 1.3%      |

Table 17: Volumetric specific heat benchmark measurement results for SS316

Extruded polystyrene foam, XPS, is a well-characterized insulation with published thermal conductivity from the National Institute of Standards and Testing [26] for temperatures between 4K and 300K [26]. Thermal conductivity is also available as a linear function of temperature for an XPS density of 32.0 kgm<sup>-3</sup>, which was used to calculate the benchmark values at 295K, and 253K [27]. The NIST value was used to calibrate the TPS 1500 at 85K explained in detail within Section 2.4. The average measured thermal conductivity of XPS at 295K, 253K, and 85K and relative absolute difference from the benchmark values are shown in Table 18. The measured thermal conductivity of XPS decreases with temperature from 29 mWm<sup>-1</sup>K<sup>-1</sup> at 295K to 24 mWm<sup>-1</sup>K<sup>-1</sup> at 253K until 10 mWm<sup>-1</sup>K<sup>-1</sup> at 85K. The RSD of measured thermal conductivity for XPS was below the uncertainty level of the TPS 1500 at 85K, 253K, and 295K. The relative absolute difference of thermal conductivity for XPS is also noticeably higher at 253K than at the other temperatures. XPS thermal conductivity measurements at 85K resulted in a 2.1% difference from the benchmark value, XPS thermal conductivity measurement results demonstrate the capability of the TPS 1500 to measure thermal conductivity of low-density, high insulating materials within the specified temperature range as well as below the minimum recommended temperature of 113K.

| Temperature | Number of<br>Measurements | AveragekBenchmarkMeasured kRSDkI    |      | Relative<br>Absolute<br>Difference  |      |
|-------------|---------------------------|-------------------------------------|------|-------------------------------------|------|
| [K]         | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]  | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]  |
| 295         | 11                        | $0.029 \pm 0.000$                   | 0.0% | 0.030                               | 2.6% |
| 253         | 10                        | $0.024\pm0.000$                     | 0.0% | 0.026                               | 5.2% |
| 85          | 8                         | $0.010\pm0.000$                     | 0.0% | 0.010                               | 2.1% |

Table 18: Thermal conductivity benchmark measurement results for XPS foam.

The measured thermal diffusivity and calculated volumetric specific heat of XPS at each measurement temperature were extracted simultaneously from the thermal conductivity measurements. The average measured thermal diffusivity and RSD for XPS at 295K, 253K, and 85K are shown in Table 19. The relative absolute difference for the measured thermal diffusivity of XPS was calculated by dividing the benchmark value for thermal conductivity by the benchmark value for volumetric specific heat using  $\rho C = \frac{k}{\alpha}$ 

(8). The average measured thermal diffusivity of XPS decreases from a value of  $0.699 \text{ mm}^2\text{s}^{-1}$  at 295K to  $0.639 \text{ mm}^2\text{s}^{-1}$  at 253K followed by an increase to  $0.751 \text{ mm}^2\text{s}^{-1}$  at 85K. The RSD of the measured thermal diffusivity at 253K is four times higher than at 295K. The relative absolute difference is above 10% for all temperatures, but the method and measurements are still considered accurate for 253K and 295K based on the validation results and specifications of the TPS 1500.

| Temperature | Number of<br>Measurements | Average Measured $\alpha$          | α<br>RSD | Benchmark<br>α                     | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|------------------------------------|----------|------------------------------------|------------------------------------|
| [K]         | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]      | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]                                |
| 295         | 11                        | $0.699\pm0.009$                    | 1.2%     | 0.802                              | 12.8%                              |
| 253         | 10                        | $0.639\pm0.032$                    | 4.9%     | 0.789                              | 19.0%                              |
| 85          | 8                         | $0.751\pm0.007$                    | 0.9%     | 0.640                              | 17.3%                              |

Table 19: Thermal diffusivity benchmark measurement results for XPS foam.

The average calculated volumetric specific heat and RSD values for XPS at 295K, 253K, and 85K are listed in Table 20. The benchmark volumetric specific heat shown in Table 20 is for an XPS density of 32.0 kgm<sup>-3</sup>, obtained from NIST, similar to the measured XPS density of 30.4 kgm<sup>-3</sup> [26]. The average calculated volumetric specific heat of XPS decreases as temperature decreases with values of 0.042, 0.038, and 0.013 MJm<sup>-3</sup>K<sup>-1</sup> at 295K, 253K, and 85K respectively. The RSD of calculated volumetric specific heat at 253K is over two times the RSD of calculated volumetric specific heat at 295K and 85K. Altogether, the variation in RSD for measured thermal conductivity, thermal diffusivity, and calculated volumetric specific heat between 85K, 253K, and 295K is comparable. The relative absolute difference for calculated volumetric specific heat is higher than 10% at all temperatures. Nevertheless, as mentioned in Section 2.2.1, XPS is known to have variations in material properties such as density and porosity depending on the preparation, which can result in larger variations between the measured thermal properties and the thermal properties available in the literature.

| Temperature | Number of<br>Measurements | Average<br>Calculated ρC             | ρC<br>RSD | Benchmark<br>ρC                      | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|--------------------------------------|-----------|--------------------------------------|------------------------------------|
| [K]         | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                                |
| 295         | 11                        | $0.042\pm0.000$                      | 1.1%      | 0.037                                | 12%                                |
| 253         | 10                        | $0.038 \pm 0.001$                    | 3.2%      | 0.032                                | 17%                                |
| 85          | 8                         | $0.013 \pm 0.000$                    | 1.2%      | 0.016                                | 13%                                |

Table 20: Volumetric specific heat benchmark measurement results for XPS foam.

Volumetric specific heat was measured independently for XPS at 85K, 253K, and 295K using the setup described in Section 2.1.3. The measured volumetric specific heat results for XPS are compared to the average calculated volumetric specific heat and the benchmark volumetric specific heat for each temperature in Table 21. T The measured volumetric specific heat for XPS is 0.030 MJm<sup>-3</sup>K<sup>-1</sup> at 295K then decreases by over 50% to 0.014 MJm<sup>-3</sup>K<sup>-1</sup> at 85K. The RSD for measured volumetric specific heat of XPS is higher than RSD for the calculated volumetric specific heat of XPS at 85K and 295K. The relative absolute differences for the average measured volumetric specific heat of XPS is comparable to the relative absolute difference for the average calculated volumetric specific heat of XPS at 85K and 295K. The measured volumetric specific heat at 295K has a large sample deviation that discouraged any further measurements at a neighboring temperature. As a result, volumetric specific heat was not measured at 253K. The measured volumetric specific heat at 85K produced an RSD and relative absolute difference lower than room temperature measurements. A potential cause for the lower measurement statistics at 85K could be the temperature stability due to the constant phase change of the liquid nitrogen as it vaporizes.

| Temperature | Type of ρC<br>Result | Number of<br>Measurements | ρC                                   | ρC<br>RSD | Relative<br>Absolute<br>Difference |
|-------------|----------------------|---------------------------|--------------------------------------|-----------|------------------------------------|
| [K]         |                      | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]       | [%]                                |
| 295         | Benchmark            | Not Available             | 0.037                                | -         | -                                  |
|             | Measured             | 10                        | $0.030\pm0.004$                      | 12%       | 19%                                |
|             | Calculated           | 11                        | $0.042\pm0.000$                      | 0.0%      | 12%                                |
| 85          | Benchmark            | Not Available             | 0.015                                | -         | -                                  |
|             | Measured             | 8                         | $0.014 \pm 0.001$                    | 3.8%      | 12%                                |
|             | Calculated           | 8                         | $0.013\pm0.000$                      | 0.0%      | 13%                                |

Table 21: Measured volumetric specific heat benchmark results for XPS.

Paraffin wax was used as a benchmark for thermal conductivity measurements because paraffin wax has a thermal conductivity value of  $0.25 \text{ Wm}^{-1}\text{K}^{-1}$  at room temperature [24]. The thermal conductivity of paraffin wax is comparable to the manufacturer's value for the thermal conductivity of RTV-655 at room temperature, 0.17 Wm<sup>-1</sup>K<sup>-1</sup>. The benchmark thermal conductivity measurements of paraffin wax were limited to the temperature of 295K due to the absence of published paraffin wax thermal conductivity values at different temperatures. The average measured thermal conductivity of paraffin wax at 295K is 0.260 Wm<sup>-1</sup>K<sup>-1</sup> with an RSD of 1.1% listed in Table 22. The relative absolute difference for the average measured thermal conductivity of paraffin wax at 295K is 4.2% when compared to the benchmark thermal conductivity value.
| Temperature | Number of<br>Measurements | Average<br>Measured k               | k<br>RSD | Benchmark<br>k                      | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|-------------------------------------|----------|-------------------------------------|------------------------------------|
| [K]         | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]                                |
| 295         | 18                        | $0.260 \pm 0.003$                   | 1.1%     | 0.250                               | 4.2%                               |

Table 22: Thermal conductivity benchmark results using paraffin wax at 295K.

The measured thermal diffusivity for paraffin wax at 295K was obtained from the thermal conductivity results. The average measured thermal diffusivity of paraffin wax at 295K is 0.119 mm<sup>2</sup>s<sup>-1</sup> with an RSD of 0.6% listed in Table 23. The average measured thermal diffusivity of paraffin wax at 295K is an order of magnitude higher than the average measured thermal diffusivity of XPS at 295K and an order of magnitude lower than the average measured thermal diffusivity of SS316 at 295K. The average measured thermal diffusivity resulted in a relative absolute difference of 7.4% when compared to the benchmark thermal diffusivity at 295K.

| Temperature | Number of<br>Measurements | Average<br>Measured α              | α<br>RSD | Benchmark<br>α                     | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|------------------------------------|----------|------------------------------------|------------------------------------|
| [K]         | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]      | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]                                |
| 295         | 18                        | $0.119\pm0.004$                    | 0.6%     | 0.111                              | 7.4%                               |

Table 23: Thermal diffusivity benchmark measurement results for paraffin wax at 295K.

The average calculated volumetric specific heat of paraffin wax at 295K was obtained from the thermal conductivity results and compared to the benchmark value for volumetric specific heat of paraffin wax at room temperature. The volumetric specific heat benchmark value was obtained from a documented measurement of paraffin wax volumetric specific heat using the TPS method available in Th [23]. The average calculated volumetric specific heat of paraffin wax is 2.184 MJm<sup>-3</sup>K<sup>-1</sup> at 295K with an RSD of 2.7% as shown in Table 24. The average calculated volumetric specific heat resulted in a relative absolute difference of 2.9% when compared to the benchmark value.

295K. Relative Benchmark Number of Average ρC RSD Absolute **Temperature Measurements** Calculated  $\rho C$ DС Difference  $[MJm^{-3}K^{-1}]$ [%]  $[MJm^{-3}K^{-1}]$ [K] [#] [%]

 $2.184 \pm 0.059$ 

2.7%

2.250

2.9%

Table 24: Calculated volumetric specific heat benchmark results for paraffin wax at

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Paraffin wax was also used to benchmark the volumetric specific heat measurement setup and method. The average measured, average calculated, and benchmark volumetric specific heats are listed for comparison in Table 25. The average measured volumetric specific heat of paraffin wax at 295K is 2.254 MJm<sup>-3</sup>K<sup>-1</sup> with an RSD of 1.7%. The average measured volumetric specific heat of paraffin wax at 295K produces a 0.2% relative absolute difference when compared to the benchmark value. The RSD and relative absolute difference are lower in the measured volumetric specific heat results are lower than the calculated volumetric specific heat results. The paraffin wax study illustrates that the experiment uncertainty can be reduced for homogeneous materials by measuring the volumetric specific heat.

| Temperature | Type of ρ <i>C</i><br>Result | Number of<br>Measurements | ρC                                   | ρC<br>RSD | Relative<br>Absolute<br>Difference |
|-------------|------------------------------|---------------------------|--------------------------------------|-----------|------------------------------------|
| [K]         |                              | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]       | [%]                                |
|             | Benchmark                    | Not Available             | 2.250                                | -         | -                                  |
| 295         | Measured                     | 11                        | $2.254\pm0.039$                      | 1.7%      | 0.2%                               |
|             | Calculated                   | 18                        | $2.184\pm0.059$                      | 2.7%      | 2.9%                               |

Table 25: Measured volumetric specific heat benchmark results for paraffin wax at 295K.

The thermal conductivity of RTV-655 is published by the manufacturer to be 0.17 Wm<sup>-1</sup>K<sup>-1</sup> at room temperature. Therefore, RTV-655 was used as a fourth benchmark for thermal conductivity measurements at 295K. The average measured thermal conductivity for RTV-655 at 295K is 0.167 Wm<sup>-1</sup>K<sup>-1</sup> with an RSD below the uncertainty of the machine as shown in Table 26. The average measured thermal conductivity of RTV-655 at 295K produces a relative absolute difference of 1.7% when compared to the benchmark thermal conductivity of RTV-655 at 295K. The benchmark results demonstrate the TPS 1500 can measure thermal conductivity of one material of interest, RTV-655.

| Temperature | Number of<br>Measurements | Average<br>Measured k               | k<br>RSD | Benchmark<br>k                      | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|-------------------------------------|----------|-------------------------------------|------------------------------------|
| [K]         | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]                                |
| 295         | 10                        | $0.167\pm0.000$                     | 0.0%     | 0.170                               | 1.7%                               |

Table 26: Thermal conductivity validation results using RTV-655 at 295K.

The average measured thermal diffusivity of RTV-655 at 295K, obtained from the thermal conductivity measurement results, is shown in Table 27. The average measured thermal diffusivity of RTV-655 at 295K is 0.108 mm<sup>2</sup>s<sup>-1</sup>, which is close in magnitude to the average measured thermal diffusivity of paraffin wax at 295K, 0.119 mm<sup>2</sup>s<sup>-1</sup>. The average measured thermal diffusivity of RTV-655 at 295 produces a relative absolute difference of 3.2% when compared to the benchmark thermal diffusivity.

| Temperature | Number of<br>Measurements | Average<br>Measured α              | α RSD | Benchmark<br>α                     | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|------------------------------------|-------|------------------------------------|------------------------------------|
| [K]         | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]   | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]                                |
| 295         | 10                        | $0.108\pm0.001$                    | 0.7%  | 0.112                              | 3.2%                               |

Table 27: Measured thermal diffusivity for RTV-655 at 295K

The calculated volumetric specific heat results for RTV-655 at 295K, obtained from the thermal conductivity measurement results, are shown in Table 28. The average calculated volumetric specific heat for RTV-655 at 295K is 1.541 MJm<sup>-3</sup>K<sup>-1</sup> with an RSD of 0.5%. The benchmark value for volumetric specific heat of RTV-655 at 295K was calculated multiplying the manufacturer's documented density of RTV-655, 1040 kgm<sup>-3</sup>, by the manufacturer's documented specific heat, 1460 Jkg<sup>-1</sup>K<sup>-1</sup>, producing a volumetric specific heat value of 1.518 MJm<sup>-3</sup>K<sup>-1</sup>. The average calculated volumetric specific heat for RTV-655 at 295K produces a relative absolute difference of 1.5% when compared to the benchmark value.

| Temperature | Number of<br>Measurements | Average<br>Calculated<br>ρC          | ρC RSD | Benchmark<br>ρC                      | Relative<br>Absolute<br>Difference |
|-------------|---------------------------|--------------------------------------|--------|--------------------------------------|------------------------------------|
| [K]         | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]    | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                                |
| 295         | 10                        | $1.541\pm0.007$                      | 0.5%   | 1.518                                | 1.5%                               |

Table 28: Calculated volumetric specific heat benchmark results for RTV-655 at 295K.

RTV-655 was the third and last material measured to benchmark the volumetric specific heat measurement method. The average measured volumetric specific heat of RTV-655 at 295K is 1.513 MJm<sup>-3</sup>K<sup>-1</sup> with an RSD of 0.8%. The average measured, average calculated, and benchmark volumetric specific heat values are listed for comparison in Table 29. The average measured volumetric specific heat results produce a relative absolute difference of 0.4% when compared to the benchmark volumetric specific heat, which is 1.1% lower than the relative absolute difference for the average calculated volumetric specific heat of 1.5%. On the other hand, the RSD of volumetric specific heat decreases from 0.8% for the measured volumetric specific heat to 0.5% for the calculated volumetric specific heat.

| Temperature | Type of <i>ρC</i><br>Result | Number of<br>Measurements | ρC                                   | ρC<br>RSD | Relative<br>Absolute<br>Difference |
|-------------|-----------------------------|---------------------------|--------------------------------------|-----------|------------------------------------|
| [K]         |                             | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]       | [%]                                |
|             | Benchmark                   | Not Available             | 1.518                                | -         | -                                  |
| 295         | Measured                    | 11                        | $1.513\pm0.012$                      | 0.8%      | 0.4%                               |
|             | Calculated                  | 10                        | $1.541 \pm 0.007$                    | 0.5%      | 1.5%                               |

Table 29: Measured volumetric specific heat benchmark results for RTV-655 at 295K.

In summary, the measurement method was validated for accuracy and precision using SS316 and XPS as the reference materials described in detail within Section 3.1.1. Thermal conductivity and volumetric specific heat measurements were also benchmarked against thermal conductivity and volumetric specific heat values available in the literature. The validation and benchmark results demonstrate the uncertainties of the measured thermal conductivities are typically lower than the uncertainties of both the measured thermal diffusivity and calculated volumetric specific heat. The volumetric specific heat measurement was not validated for accuracy and precision due to the lack of reference material with standard volumetric specific heat measurement values for the exact samples being measured for validation. Overall, the benchmark results support the continued use of the TPS 1500 to measure thermal conductivity and volumetric specific heat of RTV-655 and polyimide compound materials for and under varying temperatures.

#### 3.2 Thermal Properties of an RTV-655/Polyimide Aerogel Compound

Thermal conductivity, volumetric specific heat, and thermal diffusivity were measured or calculated for the focus materials RTV-655, polyimide aerogel, and three different volume ratios of the RTV-655/polyimide aerogel compound at 313K, 295K, 253K, and 85K. RTV-655 is a silicone rubber with elastomeric properties and a low thermal conductivity; polyimide aerogel is a foam with microscopic pores giving the aerogel a low density and low thermal conductivity. The polyimide aerogel used for this study is in the form of thin flexible sheets which can be embedded in the walls of a cylindrical tank with hemispherical ends made of RTV-655. The thermal properties of three varying volume ratios were measured to determine how the amount of polyimide

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aerogel within a multi-layer RTV-655/polyimide aerogel compound affects the thermal performance of the compound. Since the thermal properties are functions of temperature, it is necessary to measure the thermal properties at or near the operating temperatures of the application of interest. Correspondingly, thermal properties of the RTV-655/polyimide aerogel compound were measured at 85K to facilitate future assessment of the compound for low temperature applications, such as cryogenic liquid storage. The thermal properties were measured at 313K, 295K, and 253K for comparative purposes and may also be useful for other applications which may benefit from the use of an RTV-655/polyimide aerogel compound.

### 3.2.1 Thermal Properties at 313 Kelvin

Thermal conductivity of RTV-655, polyimide aerogel, and three volume ratios of the RTV-655/polyimide aerogel compound were measured at 313K with the results documented in Table 30. Neither the RTV-655 nor the polyimide aerogel have thermal property reference values at 313K. The three volume ratios of the RTV-655/polyimide aerogel compound are identified as VR20, VR34, VR56 such that the letters VR mean volume ratio and the numbers represent the targeted volume ratios of 17%, 34%, and 51%. The TPS 1500 manufacturer recommends obtaining a similar probing depth in measurements of different volume ratios of the compound [31]. First, the thermal conductivity of RTV-655 and polyimide aerogel were measured separately, at every temperature to identify the limits for thermal conductivity of VR0.0 (RTV-655) was measured 11 times, yielding an average thermal conductivity of 0.163 mWm<sup>-1</sup>K<sup>-1</sup> about 5% less than the RTV-655 manufacturer's value with an extremely low relative

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standard deviation of 0.1%. The average thermal conductivity of the polyimide aerogel sheets is 45.2 mWm<sup>-1</sup>K<sup>-1</sup>, over 70% less than the average measured thermal conductivity of RTV-655. The polyimide aerogel results also demonstrated a repeatability of 0.2%. Polyimide aerogel thermal conductivity was also measured in previous work using the TPS method with a mean result of 39.7 mWm<sup>-1</sup>K<sup>-1</sup> [14]. Thermal conductivity of the compound samples steadily declines as volume ratio increases with mean values of 0.131, 0.083, and 0.054 mWm<sup>-1</sup>K<sup>-1</sup> for VR20, VR34, and VR56, respectively. The uncertainty for all three volume ratios is 0.3% or less. Figure 23 depicts thermal conductivity for each volume ratio.

| Material        | Number of<br>Measurements | Average<br>Measured <i>k</i>        | k RSD |
|-----------------|---------------------------|-------------------------------------|-------|
|                 | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]   |
| VR0.0 (RTV-655) | 11                        | $0.163 \pm 0.000$                   | 0%    |
| VR20            | 10                        | $0.131\pm0.000$                     | 0%    |
| VR34            | 11                        | $0.083\pm0.000$                     | 0%    |
| VR56            | 11                        | $0.054\pm0.000$                     | 0%    |
| VR1.0 (PI)      | 11                        | $0.045\pm0.000$                     | 0%    |

Table 30: Thermal conductivity for the compound material measured at 313K.



Figure 23: Thermal conductivity of the compound material at 313K.

As mentioned in the validation results, thermal conductivity and thermal diffusivity of a material are measured simultaneously, which are subsequently used to calculate volumetric specific heat. Thermal diffusivity results for the RTV-655/polyimide aerogel compound at 313K, are presented in Table 31. Unlike the behavior of thermal conductivity as the volume ratio increases, thermal diffusivity decreases from RTV-655 to VR20 then increases to a thermal diffusivity of 0.33 mm<sup>2</sup>s<sup>-1</sup> for polyimide aerogel. The relative standard deviation of thermal diffusivity at 313K is higher than that of thermal conductivity at 313K, but the measurement uncertainty in terms of the relative standard deviation is below 2%. The thermal diffusivity in terms of volume ratio is depicted in Figure 24.

| Material        | Number of<br>Measurements | Average<br>Measured α              | α RSD |
|-----------------|---------------------------|------------------------------------|-------|
|                 | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]   |
| VR0.0 (RTV-655) | 11                        | $0.104\pm0.000$                    | 0.0%  |
| VR20            | 10                        | $0.066\pm0.001$                    | 1.3%  |
| VR34            | 11                        | $0.077\pm0.001$                    | 1.1%  |
| VR56            | 11                        | $0.137 \pm 0.002$                  | 1.7%  |
| VR1.0 (PI)      | 11                        | $0.330\pm0.005$                    | 1.6%  |

Table 31: Thermal diffusivity for the compound material at 313K.



Figure 24: Thermal diffusivity of the compound material at 313K.

The average calculated volumetric specific heat at 313K is listed in Table 32 for all volume ratios. The average calculated volumetric specific heat for RTV-655 of 1.57  $MJm^{-3}K^{-1}$  increases to 1.98  $MJm^{-3}K^{-1}$  for VR20 followed by a steady decline as volume ratio increases to a value of 0.14  $MJm^{-3}K^{-1}$  for polyimide aerogel. The uncertainty of the results for all materials is 2% or less.

| Material        | Number of<br>Measurements | Average<br>Calculated<br>ρC          | Calculated<br>ρC RSD |
|-----------------|---------------------------|--------------------------------------|----------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                  |
| VR0.0 (RTV-655) | 11                        | $1.572\pm0.007$                      | 0.5%                 |
| VR20            | 10                        | $1.975\pm0.024$                      | 1.2%                 |
| VR34            | 11                        | $1.071\pm0.013$                      | 1.2%                 |
| VR56            | 11                        | $0.396\pm0.008$                      | 2.0%                 |
| VR1.0 (PI)      | 11                        | $0.137\pm0.002$                      | 1.5%                 |

Table 32: Volumetric specifc heat for the compound 313K

Volumetric specific heat was measured separately for RTV-655, polyimide aerogel, and the three volume ratios of the compound at 313K. RTV-655 was measured first and resulted in a mean value of 1.531 MJm<sup>-3</sup>K<sup>-1</sup>, higher than the room temperature reference value. As shown in Table 33, the average measured volumetric specific heat decreases as volume ratio increases. The uncertainty of the measurements at 313K was lower than the uncertainty at other temperatures and is likely due to the ability of the environmental chamber heating mode to maintain a constant ambient temperature. A comparison between the measured and volumetric specific heat, is shown in Figure 25. Despite the differences between the volume ratios used for the calculated and measured volumetric specific heat measurements, it can inferred from Figure 25 that the measured volumetric specific heat values for the heterogeneous materials, (0>VR>1), are significantly different from the calculated volumetric specific heat values when compared to the differences for the homogeneous materials, RTV-655 (VR=0), and polyimide aerogel (VR=1).

| Material        | Number of<br>Measurements | Average Measured $\rho C$            | Measured<br>ρC RSD |
|-----------------|---------------------------|--------------------------------------|--------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                |
| VR0.0 (RTV-655) | 10                        | $1.671\pm0.005$                      | 0.3%               |
| VR20            | 10                        | $1.531\pm0.012$                      | 0.8%               |
| VR47            | 10                        | $1.267\pm0.004$                      | 0.3%               |
| VR71            | 10                        | $1.106\pm0.009$                      | 0.8%               |
| VR1.0 (PI)      | 11                        | $0.253 \pm 0.004$                    | 1.6%               |

Table 33: Measured volumetric specific heat of the compound material at 313K.



Figure 25: Comparison of measured and calculated volumetric specific heat at 313K.

## 3.2.2 Thermal Properties at 295 Kelvin

Thermal conductivity for RTV-655, polyimide aerogel sheets and three volume ratios of the RTV-655/polyimide aerogel compound were measured at 295K. The average measured thermal conductivity and average sample standard deviation for each volume

ratio of the compound material at 295K are shown in Table 34. The average measured thermal conductivity of RTV-655 is  $0.167 \text{ Wm}^{-1}\text{K}^{-1}$ , 1.7% lower than the manufacturer's value of  $0.17 \text{ Wm}^{-1}\text{K}^{-1}$ . The polyimide aerogel has an average measured thermal conductivity of 48.4 mWm<sup>-1</sup>K<sup>-1</sup>. The average measured thermal conductivities for the three volume ratios decreased as volume ratio increased with values of 0.122, 0.082, and 0.053 Wm<sup>-1</sup>K<sup>-1</sup> for VR20, VR34, and VR56, respectively. The average measured thermal conductivity at varying volume ratios is clearly depicted in Figure 26.

| Material        | Number of<br>Measurements | Average<br>Measured <i>k</i>        | k<br>RSD |
|-----------------|---------------------------|-------------------------------------|----------|
|                 | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      |
| VR0.0 (RTV-655) | 10                        | $0.167\pm0.001$                     | 0.3%     |
| VR20            | 10                        | $0.122\pm0.002$                     | 1.4%     |
| VR34            | 10                        | $0.082\pm0.000$                     | 0.0%     |
| VR56            | 10                        | $0.053 \pm 0.000$                   | 0.0%     |
| VR1.0 (PI)      | 10                        | $0.048 \pm 0.001$                   | 1.7%     |

Table 34: Thermal Conductivity for the compound material at 295K.



Figure 26: Thermal conductivity of the compound material at 295K.

Thermal diffusivity measurements, which were obtained from the thermal conductivity measurements of the compound at 295K, are presented in Table 35. The variation in the average measured thermal diffusivity for different volume ratios at 295K is comparable to the variation observed at 313K. The average measured thermal diffusivity decreases from RTV-655 to VR34, and then increases to polyimide aerogel. Figure 27 depicts the average measured thermal diffusivity of the RTV-655/polyimide aerogel as volume ratio changes at 295K. The relative standard deviation for measured thermal diffusivity for VR20 was highest at 10% as compared to the other volume ratios.

| Material        | Number of<br>Measurements | Average Measured $\alpha$          | α RSD |
|-----------------|---------------------------|------------------------------------|-------|
|                 | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]   |
| VR0.0 (RTV-655) | 10                        | $0.108\pm0.001$                    | 0.7%  |
| VR20            | 10                        | $0.080\pm0.008$                    | 10.0% |
| VR34            | 10                        | $0.078 \pm 0.001$                  | 0.8%  |
| VR56            | 10                        | $0.129 \pm 0.005$                  | 4.1%  |
| VR1.0 (PI)      | 10                        | $0.293\pm0.017$                    | 5.7%  |

Table 35: Thermal diffusivity for all materials measured at 295K.



Figure 27: Thermal diffusivity for varying volume ratios of the compound material at 295K.

Volumetric specific heat was calculated from the measured thermal conductivity and thermal diffusivity of the compound at 295K and the results are listed in Table 36. The difference in the average calculated volumetric specific heat at 295K is statistically insignificant between RTV-655 and VR20. For volume ratios greater than VR20, the average calculated volumetric specific heat decreases as volume ratio increases. The average calculated volumetric specific heat for varying volume ratios is shown in Figure

28 Error! Reference source not found.at 295K.

| Material        | Number of<br>Measurements | Average<br>Calculated ρC             | Calculated $\rho C \text{ RSD}$ |
|-----------------|---------------------------|--------------------------------------|---------------------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                             |
| VR0.0 (RTV-655) | 10                        | $1.541\pm0.007$                      | 0.5%                            |
| VR20            | 10                        | $1.537\pm0.143$                      | 9.3%                            |
| VR34            | 10                        | $1.055\pm0.011$                      | 1.0%                            |
| VR56            | 10                        | $0.413 \pm 0.015$                    | 3.6%                            |
| VR1.0 (PI)      | 10                        | $0.166\pm0.007$                      | 4.1%                            |

Table 36: Calculated volumetric specific heat for the compound at 295K.



Figure 28: Calculated volumetric specific heat for the RTV-655/polyimide aerogel compoundat 295K.

Volumetric specific heat at 295K was measured for RTV-655, polyimide aerogel, and three volume ratios of the compound recorded in Table 37. The volumetric specific

heat of RTV-655 was measured as a validation material at 295K as described in Section 3.1.1. The average measured volumetric specific heat for VR20, VR47, and VR71 are 1.494, 1.265, 1.096 MJm<sup>-3</sup>K<sup>-1</sup>, respectively. The larger uncertainty values at 295K as compared to the uncertainty values at 313K are likely attributable to the larger variations in the ambient conditions. As mentioned in Section 2.3.2.2, the 295K experiments were performed using the ambient conditions of the laboratory, as compared to the more controlled temperature conditions attainable in the environmental chamber. Results of the volumetric specific heat measurements at varying volume ratios at 295K are shown in Table 37. Similar to measurements at 313K, larger differences in measured and calculated volumetric specific heat values for the heterogeneous compound materials are apparent when compared to the homogeneous RTV-655 and polyimide aerogel, illustrated in Figure 29.

| Material        | Number of<br>Measurements | Average<br>Measured ρ <i>C</i>       | Measured<br>ρC RSD |
|-----------------|---------------------------|--------------------------------------|--------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                |
| VR0.0 (RTV-655) | 11                        | $1.513\pm0.012$                      | 0.8%               |
| VR20            | 11                        | $1.494\pm0.076$                      | 5.1%               |
| VR34            | 11                        | $1.265\pm0.068$                      | 5.4%               |
| VR56            | 16                        | $1.096\pm0.063$                      | 5.7%               |
| VR1.0 (PI)      | 36                        | $0.275 \pm 0.022$                    | 7.9%               |

Table 37: Measured volumetric specific heat of the compound at 295K.



Figure 29: Comparison of measured and calculated volumetric specific heat at 295K.

#### 3.2.3 Thermal Properties at 253 Kelvin

Thermal conductivity for RTV-655, polyimide aerogel sheets and three volume ratios of the RTV-655/polyimide aerogel compound were measured at 253K with the results documented in Table 38. The average measured thermal conductivity of RTV-655 is 0.168 Wm<sup>-1</sup>K<sup>-1</sup> at 253K, 1 mWm<sup>-1</sup>K<sup>-1</sup> higher than the average measured thermal conductivity of RTV-655 at 295K. The average measured thermal conductivity of the polyimide aerogel sheets is 36.0 mWm<sup>-1</sup>K<sup>-1</sup> at 253K, a decrease of 34% from the average measured thermal conductivity of the polyimide aerogel sheets at 295K. The average measured thermal conductivity of the polyimide aerogel sheets at 295K. The average measured thermal conductivity of the polyimide aerogel sheets at 295K. The average measured thermal conductivities for the three volume ratios decreased as volume ratio increased with values of 0.108, 0.073, and 0.049 Wm<sup>-1</sup>K<sup>-1</sup> for VR20, VR34, and VR56, respectively. The measured sample standard deviations for the 253K measurements is at least one order of magnitude higher than most measurements at different temperatures. Figure 30. shows the average measured thermal conductivity along with vertical sample

standard deviation bars for each volume ratio at 253K. The increased measured sample standard deviation at 253K area result of the variability in ambient conditions within the chest freezer described in Section 2.3.1.3.

| Material        | Number of<br>Measurements | Average<br>Measured <i>k</i>        | k<br>RSD |
|-----------------|---------------------------|-------------------------------------|----------|
|                 | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      |
| VR0.0 (RTV-655) | 11                        | $0.168 \pm 0.005$                   | 3.0%     |
| VR20            | 9                         | $0.108\pm0.005$                     | 4.5%     |
| VR34            | 10                        | $0.073 \pm 0.006$                   | 7.9%     |
| VR56            | 14                        | $0.049 \pm 0.003$                   | 7.0%     |
| VR1.0 (PI)      | 17                        | $0.036 \pm 0.000$                   | 1.1%     |

Table 38: Average measured thermal conductivity for the compound at 253K.



Figure 30: Measured thermal conductivity for varying volume ratios the compound at 253K.

Thermal diffusivity was obtained from the thermal conductivity measurements at 253K, listed in Table 39. The variation in the average measured thermal diffusivity for different volume ratios at 253K is comparable to the variation observed at 295K and 313K. The average measured thermal diffusivity decreases from RTV-655 to VR20, and then increases to polyimide aerogel. Figure 31 illustrates the average measured thermal diffusivity of the RTV-655/polyimide aerogel as volume ratio changes at 253K. The relative standard deviation for the thermal diffusivity measurements at 253K is the highest for VR20 and VR34 at 33.6% and 32.7%, respectively, compared to the other volume ratios. Figure 31 illustrates the variation in the measured thermal diffusivity for varying volume ratios of the RTV-655/polyimide aerogel compound at 253K.

| Material        | Number of<br>Measurements | Average<br>Measured α              | a RSD |
|-----------------|---------------------------|------------------------------------|-------|
|                 | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]   |
| VR0.0 (RTV-655) | 11                        | $0.117\pm0.023$                    | 19%   |
| VR20            | 9                         | $0.031\pm0.011$                    | 34%   |
| VR34            | 10                        | $0.083\pm0.027$                    | 33%   |
| VR56            | 14                        | $0.105\pm0.015$                    | 14%   |
| VR1.0 (PI)      | 17                        | $0.311\pm0.016$                    | 5%    |

Table 39: Thermal diffusivity of the compound material at 253K.



Figure 31: Thermal diffusivity of the compound material at 253K.

Volumetric specific heat was calculated from the measured thermal conductivity and thermal diffusivity of the compound at 253K and the results are recorded in Table 40. The variation in the average calculated volumetric specific heat at 253K is similar to variations observed at 295K and 313K. The average calculated volumetric specific heat values increases from RTV-655 to VR20 followed by a decrease to the value for the polyimide aerogel sheets. The relative standard deviation of the calculated volumetric specific heat is the highest for VR20 and VR34, which agrees with the measured thermal conductivity and measured thermal diffusivity results.

| Material        | Number of<br>Measurements | Average<br>Calculated<br>ρC          | Calculated $\rho C RSD$ |
|-----------------|---------------------------|--------------------------------------|-------------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                     |
| VR0.0 (RTV-655) | 11                        | $1.477\pm0.253$                      | 17%                     |
| VR20            | 9                         | $3.793 \pm 1.110$                    | 29%                     |
| VR34            | 10                        | $0.933 \pm 0.233$                    | 25%                     |
| VR56            | 14                        | $0.477\pm0.047$                      | 9.9%                    |
| VR1.0 (PI)      | 17                        | $0.116\pm0.005$                      | 4.5%                    |

Table 40: Volumetric specific heat for the compound at 253K.

Volumetric specific heat at 253K was measured for RTV-655, polyimide aerogel, and three volume ratios of the compound with the results shown in Table 41. RTV-655 was the first material measured resulting in an average value of 1.477 MJm<sup>-3</sup>K<sup>-1</sup>, lower than the average measured volumetric specific heat at 295K. The average measured volumetric specific heat for the three volume ratios decreases as volume ratio increases. The relative standard deviation is the lowest for RTV-655, but the relative standard deviation of the polyimide aerogel measurements is an order of magnitude higher than RTV-655 and the three volume ratios. A comparison of measured and calculated volumetric specific heat at 253K is shown in Figure 32. As observed at 313K and 293K, larger differences in measured and calculated volumetric specific heat values for the heterogeneous compound materials (0>VR>1) are apparent when compared to the homogeneous RTV-655 (VR=0) and polyimide aerogel (VR=1).

| Material        | Number of<br>Measurements | Average Measured $\rho C$            | Measured<br>ρC RSD |
|-----------------|---------------------------|--------------------------------------|--------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                |
| VR0.0 (RTV-655) | 7                         | $1.477\pm0.003$                      | 0.2%               |
| VR20            | 6                         | $1.444\pm0.107$                      | 7.4%               |
| VR47            | 6                         | $1.190\pm0.089$                      | 7.5%               |
| VR71            | 5                         | $1.000\pm0.073$                      | 7.3%               |
| VR1.0 (PI)      | 12                        | $0.258 \pm 0.052$                    | 20.2%              |

Table 41: Measured volumetric specific heat of the compound at 253K.



Figure 32: Illustration of the compound material volumetric specific heat changes with volume ratio.

# 3.2.4 Thermal Properties at 85 Kelvin

Thermal conductivity for RTV-655, polyimide aerogel sheets and three volume ratios of the RTV-655/polyimide aerogel compound were measured at 85K. XPS insulation and its reference thermal conductivity at 85K were used to calibrate the TCR

value of 0.014373 K<sup>-1</sup>, which was described in Section 2.4. The average measured thermal conductivity for RTV-655, polyimide aerogel, and the three volume ratios are 25% lower at 85K from 295K. The variation of the average measured thermal conductivity of the RTV-655/polyimide aerogel compound material is comparable to that at 253K, 295K, and 313K. The average measured thermal conductivities for the three volume ratios at 85K decreased as volume ratio increased with values of 0.091, 0.065, and 0.029 Wm<sup>-1</sup>K<sup>-1</sup> for VR20, VR34, and VR56, respectively. The average measured thermal conductivity and the sample standard deviation at varying volume ratios is shown in Figure 33.

| Material        | Number of<br>Measurements | Average<br>Measured k               | k<br>RSD |
|-----------------|---------------------------|-------------------------------------|----------|
|                 | [#]                       | [Wm <sup>-1</sup> K <sup>-1</sup> ] | [%]      |
| VR0.0 (RTV-655) | 25                        | $0.113 \pm 0.003$                   | 2.8%     |
| VR20            | 17                        | $0.091\pm0.001$                     | 1.0%     |
| VR34            | 17                        | $0.065\pm0.004$                     | 5.5%     |
| VR56            | 26                        | $0.029 \pm 0.000$                   | 0.0%     |
| VR1.0 (PI)      | 40                        | $0.014 \pm 0.001$                   | 9.4%     |

Table 42: Thermal conductivity for the compound at 85K.



Figure 33. Thermal conductivity of the compound at 85K.

Thermal diffusivity measurements, which were obtained from the thermal conductivity measurements of the compound at 295K, are presented in Table 43. The variation in the average measured thermal diffusivity for different volume ratios at 85K is comparable to the variations observed at 253K, 295K, and 313K. The average measured thermal diffusivity decreases in value from RTV-655 to VR20, and then increases to the value for to polyimide aerogel. Figure 34: Thermal diffusivity of the compound at 85K... depicts the average measured thermal diffusivity of the RTV-655/polyimide aerogel as volume ratio changes at 85K. The relative standard deviation for measured thermal diffusivity for VR56 and the polyimide aerogel sheets are the highest at 32.5% and 33.2%, respectively, as compared to the other volume ratios.

| Material        | Number of<br>Measurements | Average<br>Measured α              | α RSD |
|-----------------|---------------------------|------------------------------------|-------|
|                 | [#]                       | [mm <sup>2</sup> s <sup>-1</sup> ] | [%]   |
| VR0.0 (RTV-655) | 25                        | $0.386 \pm 0.013$                  | 3.5%  |
| VR20            | 17                        | $0.061\pm0.006$                    | 10.0% |
| VR34            | 17                        | $0.062\pm0.009$                    | 14.8% |
| VR56            | 26                        | $0.141 \pm 0.046$                  | 32.5% |
| VR1.0 (PI)      | 40                        | $0.067\pm0.022$                    | 33.2% |

Table 43: Thermal diffusivity for the compound at 85K.



Figure 34: Thermal diffusivity of the compound at 85K.

Volumetric specific heat was calculated from the measured thermal conductivity and measured thermal diffusivity results for the RTV-655/polyimide aerogel compound at 85K, with the results recorded in Table 44. The average calculated volumetric specific heat at 85K increases in value from RTV-655 to VR20 followed by a decrease in value as volume ratio increases to VR1.0.

| Material        | Number of<br>Measurements | Average<br>Calculated<br>ρC          | Calculated<br>ρC RSD |
|-----------------|---------------------------|--------------------------------------|----------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                  |
| VR0.0 (RTV-655) | 25                        | $0.294\pm0.018$                      | 6.1%                 |
| VR20            | 17                        | $1.495\pm0.149$                      | 9.9%                 |
| VR34            | 17                        | $1.084\pm0.244$                      | 23%                  |
| VR56            | 26                        | $0.227\pm0.079$                      | 35%                  |
| VR1.0 (PI)      | 40                        | $0.234\pm0.092$                      | 39%                  |

Table 44: Calculated volumetric specific heat at 85K.

Volumetric specific heat at 85K was measured for RTV-655, polyimide aerogel sheets, and three volume ratios of the compound as shown in Table 45. The average measured volumetric specific heat at 85K decreases in value from RTV-655 to VR34, then increases in value at VR51, followed by a decrease to the measured volumetric specific heat value of VR1. The relative standard deviation is higher for the less dense materials. A comparison between measured and calculated volumetric specific heat at

85K is shown in



Figure 29. Again, larger differences in measured and calculated volumetric specific heat values for the heterogeneous compound materials (0>VR>1) are apparent when compared to the homogeneous RTV-655 (VR=0) and polyimide aerogel (VR=1).

| Material        | Number of<br>Measurements | Average<br>Measured ρC               | Measured<br>ρC RSD |
|-----------------|---------------------------|--------------------------------------|--------------------|
|                 | [#]                       | [MJm <sup>-3</sup> K <sup>-1</sup> ] | [%]                |
| VR0.0 (RTV-655) | 8                         | $0.544 \pm 0.006$                    | 1.2%               |
| VR20            | 10                        | $0.450\pm0.026$                      | 5.7%               |
| VR47            | 11                        | $0.365\pm0.008$                      | 2.2%               |
| VR71            | 8                         | $0.464\pm0.013$                      | 2.7%               |
| VR1.0 (PI)      | 7                         | $0.075\pm0.023$                      | 31.1%              |

Table 45: Measured volumetric specific heat of the compound at 85K.





## 3.2.5 Thermal Properties Under Varying Temperature and Varying Volume Ratio

The figures presented in this section are a compilation of the results presented in Sections 3.2.1 to 3.2.4. Thermal conductivity was measured at 85K, 253K, 295K, and 313K for RTV-655, polyimide aerogel, and three volume ratios of the compound shown



Figure 37. RTV-655 exhibits the highest average thermal conductivity and polyimide aerogel has the lowest average thermal conductivity. At each temperature studied, the average thermal conductivity decreases as volume ratio increases. Generally, the thermal conductivity measurements of the homogeneous materials have lower relative standard deviations than the heterogeneous materials.



Figure 36: Thermal conductivity for varying volume ratios at each temperature.



Figure 37: Thermal conductivity for each temperature at varying volume ratios.



Figure 39 shows the variation in the average measured thermal diffusivity and sample standard deviation for each temperature at varying volume ratios. The average measured thermal diffusivity exhibits similar variability and is the lowest at VR17 and

VR34 for each temperature. Also, the uncertainties for measured thermal diffusivity and calculated volumetric specific heat are overall higher than the uncertainties observed for measured thermal conductivity. The common variation in both measured thermal diffusivity and calculated volumetric specific heat is due to the relationship between the two properties and the dependency of calculated volumetric specific heat on the measured thermal conductivity and measured thermal diffusivity, which are measured simultaneously.



Figure 38: Thermal diffusivity for varying volume ratios at each temperature.



Figure 39: Thermal diffusivity for each temperature at varying volume ratios.



Figure 40 and Figure 41 depict the average calculated volumetric specific heat behavior for varying volume ratio at each temperature. The variation in the average calculated volumetric specific heat is inverse to the variation in thermal diffusivity at each temperature and volume ratio. Moreover, the sample uncertainties for each measurement temperature and volume ratio is higher than thermal conductivity, especially for the heterogeneous materials.


Figure 40: Calculated volumetric specific heat for each temperature at each volume ratio.



Figure 41: Calculated volumetric specific heat for each volume ratio at each temperature.



Figure 43 show the average measured volumetric specific heat behavior for varying volume ratio at each temperature. The variation in measured volumetric specific heat is different from the variation in calculated volumetric specific heat. The average measured volumetric specific heat steadily decreases as volume ratio increases for all temperatures except 85K, where VR51 increases from VR34. Moreover, the uncertainties for the measured volumetric specific heat are lower than the uncertainties for the calculated volumetric specific heat with the exception of polyimide aerogel.



Figure 42: Measured volumetric specific heat for each volume ratio at varying temperatures.



Figure 43: Measured volumetric specific heat for each temperature at varying volume ratios.

#### CHAPTER 4.

### SUMMARY AND CONCLUSION

#### 4.1 Summary

Thermal conductivity and volumetric specific heat were measured for RTV-655, polyimide aerogel, and three volume ratios of the compound material. The measurements at 253K occurred within a freezer; measurements at 295K occurred in the open environment of the lab; and measurements at 313K occurred in an environmental chamber using the same sample holder for the three temperatures as well as both measurement types. The samples were measured at 85K by immersing a low temperature apparatus containing the measurement setup into liquid nitrogen. Validation measurements were performed using two reference materials and measured properties at 295K: SS316 and XPS. Following the validation of the TPS 1500, benchmark measurements were conducted using SS316, XPS, paraffin wax, and RTV-655. The validation and benchmark measurements demonstrated the capability of the TPS 1500 to measure the thermal conductivities ranging between 0.001 to 20 Wm<sup>-1</sup>K<sup>-1</sup> of materials with densities ranging from 30 to 8000 kgm<sup>-3</sup>.

The materials of focus for this study, RTV-655 and polyimide aerogel, were measured separately and at three different volume ratios. Thermal conductivity and volumetric specific heat were only documented in literature for RTV-655 around 295K. The sizes of the RTV-655 and compound samples were designed to satisfy the minimum sample size constraints for the TPS 1500, but the sample sizes of the available polyimide aerogel were below the recommended minimum sample size for the TPS 1500. As mentioned in Section 2.2.2, the polyimide samples were synthesized by NASA [13].

Thermal conductivity results contain the three properties of interest: thermal conductivity, thermal diffusivity, and volumetric specific heat.

Thermal conductivity validation measurements from 253K to 313K are within the rated accuracy of the machine and design specifications [33]. The temperature of 85K is outside the TPS 1500's designed temperature range, but the 85K measurements were attempted based on the manufacturer's recommendation by calibrating the sensor's TCR value using extruded polystyrene as the calibration material. The resulting TCR value was used for all thermal conductivity and volumetric specific heat measurements at 85K. At all temperatures, the average measured thermal conductivity decreases as volume ratio increases from VR=0 (RTV-655) to VR=1 (polyimide aerogel). The average measured thermal conductivity does not change significantly for the same material between 253K and 313K, but the average measured thermal conductivity for all materials experienced a significant decrease from 253K to 85K. The average measured thermal diffusivity does not steadily decrease with temperature. The average calculated volumetric specific heat decreases from 313K to 85K except for XPS and polyimide aerogel whose value increased from 253K to 85K. The relationship between calculated volumetric specific heat and volume ratio is consistent at each temperature measured. Specifically, as volume ratio increases the calculated volumetric specific heat increases to the value measured at VR20. Then, as volume ratio increases above VR20, the calculated volumetric specific heat decreases to the value measured for polyimide aerogel. Overall, the thermal conductivity results were accurate when compared to available reference data and the measurements were repeatable as observed in the relatively low uncertainty values determined at each temperature and volume ratio studied. However, as stated in Section

2.1.1, the volumetric specific heat of heterogeneous compounds, such as the compound RTV-655/polyimide aerogel samples, requires an independent, volumetric specific heat measurement using a separate setup over the same temperatures. Reference materials were not provided by the manufacturer for validation of the volumetric specific heat setup and measurement. Thus, the volumetric specific heat of XPS, paraffin wax, and RTV-655 was measured at 295K and benchmarked by comparing the measurements to values reported in the literature. As stated in Section 2.1.3, the results for homogeneous materials can be compared to the calculated volumetric specific heat results. Overall, the benchmark volumetric specific heat measurements yielded low uncertainty values and low relative absolute differences as compared to reference values found in the literature. The volumetric specific heat measurements at 85K utilized the same TCR value obtained from thermal conductivity measurements and yielded volumetric specific heat measurements with lower relative standard deviations than at 253K and 295K. Measured volumetric specific heat decreases as temperature decreases. Based on the uncertainties, the measured volumetric specific heat values of the homogeneous materials (RTV-655 and polyimide aerogel) and heterogeneous compounds at each temperature are statistically significant. The measured volumetric specific heat decreases as volume ratio increases for all temperatures except 85K. At 85K, the change in measured volumetric specific heat exhibits a subtle variation than that of the calculated volumetric specific heat. The differences observed between measured and calculated volumetric specific heat is in line with the recommendation provided by the manufacturer to measure volumetric specific heat independently.

### 4.2 Conclusions

The TPS method is most widely documented for its ability to measure thermal conductivity, thermal diffusivity, and volumetric specific heat. The measured volumetric specific heat setup is a derivative of the TPS capability to act as a heat source and a thermocouple. The validation experiments and the relatively small experiment uncertainties in the RTV-655, polyimide aerogel, and compound material results confirm that the transient plane source method and experiment setup employed were accurate in measuring the thermal conductivity. The larger uncertainties observed in the measured volumetric specific heat are a result of the variability in experimental setup and environment as discussed Section 3.2. Despite the larger uncertainty, a direct measurement of the volumetric specific heat was the only viable alternative using the TPS system to provide any useful volumetric specific heat data for the compound RTV-655/polyimide materials.

The lower uncertainties reported for the measured thermal conductivity as compared to the measured volumetric specific heat could be a result of several factors. To begin with, thermal conductivity is obtained in a single measurement independent of any other measurement, but measured volumetric specific heat is dependent on a reference measurement. Any differences between the reference and sample measurement such as temperature, contact between the cup and insulation, and the delamination of the sensor from the cup will infer an error into the measured volumetric specific heat results. Also, the volumetric specific heat measurement method does not have a clear-cut rule for determining measurement time of a sample. The differences in measurement time affect the measured volumetric specific heat results disproportionally, thus, making it difficult

to assess the correct measurement time. The thermal settling time is provided as a guide to estimate the measurement time. However, the thermal settling time is calculated using the measured thermal diffusivity, which is known to be less accurate, as stated in Section 2.1.1. Another source of uncertainty is determining the correct power increase from the reference to the sample measurement. The power increase should be optimized to equivocate the temperature increase in the sample measurement to the temperature increase in the reference measurement. Optimization of the power increase is challenging due to fluctuations in the sample environment, which cause variations in the temperature increase. Subsequently, the optimal power is variable and must be determined for each measurement. The difficulty in equivocating the temperature increase between the sample and reference measurements culminates in larger uncertainties for the measured volumetric specific heat results. Lastly, the TPS method for measuring volumetric specific heat is for measuring low density materials, such as XPS and polyimide aerogel. Since the amount of material that can be measured is restricted by the small fixed volume of the cup, the mass of the low-density materials in this study is an order of magnitude smaller than the amount of mass for the higher density materials. Recalling that mass and temperature normalize the amount of heat absorbed by a material resulting in the property of specific heat, it can be reasoned that the lower amount of mass of XPS and polyimide aerogel exacerbates the requisite of matching sample and reference temperature differences. Altogether, the TPS method for measuring volumetric specific heat of lowdensity, low-thermal conductivity materials provided less repeatable results despite the attempts to optimize and control the experiments. More accurate measurements of volumetric specific heat will likely require the use of a differential scanning calorimeter,

DSC. The DSC employs the same tactic as the TPS volumetric specific heat method with a comparison between reference and sample measurements, but a DSC has much better control between the reference and sample measurement environments.

The thermal conductivity results obtained herein are useful in filling the knowledge gap for RTV-655, polyimide aerogel, and the compound materials under varying temperature. For steady and quasi-steady analyses, the thermal conductivity results can be used to enhance future design and applications which may benefit from the use of these materials over a range of temperatures. Of particular interest, knowledge of the thermal conductivity of these materials at cryogenic temperatures can now be used in computational models to further study the performance of the RTV-655/polyimide co

mpounds as a viable construction material for future cryogenic propellant storage tanks in a space.

#### REFERENCES

- [1] R. Radebaugh, "About Cryogenics," *The MacMillan Encyclopedia of Chemistry*, 2002.
- [2] S. Gursu, S. A. Sherif, T. N. Veziroglu and J. W. Sheffield, "Analysis and Optimization of Thermal Stratification and Self-Pressurization Effects in Liquid Hydrogen Storage Systems--Part 1: Model Development," *Journal of Energy Resources Technology*, vol. 115, no. 3, pp. 221-227, 1993.
- [3] S. A. Dye, A. Kopelove and G. L. Mills, "Integrated and Load Responsive Multilayer Insulation," *Advances in Cryogenic Engineering: Transactions of the Cryogenic Engineering Conference*, pp. 946-953, 2010.
- [4] J. Marchetta and F. Sabri, "Development, Characterization, and Validation of an Aerogel/RTV Based Cryogenic Propellant Tank," 2015.
- [5] "Staying Cool on the ISS," 2001. [Online]. Available: https://science.nasa.gov/science-news/science-at-nasa/2001/ast21mar\_1. [Accessed 1 March 2019].
- [6] K. M. Smith, "Thermal Characterization of Polyurea Cross-Linked Silica Aerogel-RTV 655 for Cryogenic Tank Applications," Memphis, 2012.
- [7] G. W. Vogl, "Comprehensive Theory of Heat Transfer in Heterogeneous Materials," Blacksburg, 2003.
- [8] Momentive Performance Materials Inc., "RTV-655 Technical Data Sheet," Momentive Performance Materials, 24 April 2012. [Online]. Available: https://www.exdron.co.il/images/Products/files/GE-MOMENTIV-RTV-615-1-pint-500ml-datasheet.PDF.
- [9] T. M. Mower, "Thermomechanical behavior of aerospace-grade RTV (silicone adhesive)," *International Journal of Adhesion and Adhesives*, vol. 87, pp. 64-72, 2018.
- [10] M. A. Meador, "Insulation Chemistry on Earth and Beyond: Polyimide and Polyamide Aerogels," 13 July 2016. [Online]. Available: https://www.acs.org/content/dam/acsorg/events/technology-innovation/Slides/2016-07-14-aerogels-slides.pdf.
- [11] D. Williams, "A Small Scale RTV-655Aerogel Cryogenic Liquid Storage Tank for Space Applications," Memphis, TN, 2016.
- [12] H. Maleki, L. Duraes and A. Portugal, "An overview on silica aerogels synthesis and different mechanical reinforcing strategies," *Journal of Non-Crystalline Solids*, vol. 385, pp. 55-74, 2014.
- [13] H. Guo, M. A. Meador, L. McCorkle, D. J. Quade, J. Guo, B. Hamilton, M. Cakmak and G. Sprowl, "Polyimide Aerogels Cross-Linked through Amine Functionalized Polyoligomeric Silsesquioxane," ACS Applied Materials & Interfaces, pp. 546-552, 4 February 2011.
- [14] D. Pumroy, "Development of an RTV-655/Aerogel Cryogenic Liquid Storage Tank for Space Applications," Memphis, TN, 2014.

- [15] International Organization for Standardization, "Plastics--Determination of thermal conductivity and thermal diffusivity--Part2: Transient plane source (hot disc) method," 2015.
- [16] F. Sabri, J. G. Marchetta, K. M. Rifat Faysal, A. Brock and E. Roan, "Effect of Aerogel Particle Concentration on Mechanical Behavior of Impregnated RTV 655 Compound Material for Aerospace Applications," *Advances in Materials Science and Engineering*, vol. 2014, no. Article ID 216356, p. 10 pages, 15 October 2014.
- <sup>[17]</sup> ThermTest, Inc, "Illustration of the placement of the Hot Disk double sided sensor during thermal conductivity testing," 2019.
- <sup>[18]</sup> Thermtest Inc., "Materials Thermal Properties Database," 2018. [Online]. Available: https://thermtest.com/materials-database#316.
- [19] T. K. Chu and C. Y. Ho, "Electrical Resistivity and Thermal Conductivity of Nine Selected AISI Stainless Steels," CINDAS/Purdue University, West Lafayette, 1977.
- [20] P. E. Bradley and R. Radebaugh, "Properties of Selected Materials at Cryogenic Temperatures," in *CRC Handbook of Chemistry and Physics*, W. M. Haynes, Ed., CRC Press, 2016.
- [21] C. S. Kim, "Thermophysical Properties of Stainless Steels," Argonne National Laboratory, Argonne, 1975.
- [22] Thermtest Inc., "Measuring Specific Heat Capacity of Paraffin Wax Using the TPS Specific Heat Method," 7 February 2015. [Online]. Available: https://thermtest.com/measuring-specific-heat-capacity-of-paraffin-wax-using-thetps-specific-heat-method.
- [23] "Engineering ToolBox," 2003. [Online]. Available: https://www.engineeringtoolbox.com/thermal-conductivity-d\_429.html.
- [24] NIST, "Material Properties: Polystyrene," [Online]. Available: https://trc.nist.gov/cryogenics/materials/Polystyrene/polystyrenerev.html.
- [25] A. A. Abdou and I. M. Budaiwi, "Comparison of Thermal Conductivity Measurements of Building Insulation Materials under Various Operating Temperatures," *Journal of Building Physics*, vol. 29, no. 2, pp. 171-184, October 2005.
- [26] M. Koru, "Determination of Thermal conductivity of Closed-Cell Insulation Materials That Depend on Temperature and Density," King Fahd University of Petroleum & Minerals, 2016.
- [27] A. Baudot, J. Mazuer and J. Odin, "Thermal Conductivity of a RTV Silicone Elastomer Between 1.2 and 300 K," *Cryogenics*, vol. 38, no. 2, pp. 227-230, February 1998.
- <sup>[28]</sup> Thermtest Inc, 19 March 2019. [Online]. Available: https://thermtest.com/faq.
- [29] ThermTest Inc., "Tech Talk Optimizing Measurements of Low Density/High Insulating Materials using the Transient Plane source (TPS) System," 2011.
- <sup>[30]</sup> ThermTest Inc., "TPS Sensors," 2018.
- <sup>[31]</sup> ThermTest, Inc., ""Specific Heat Testing Services"," 2019.
- [32] W. S. Janna, Engineering Heat Transfer, 3 ed., Boca Raton, Florida: CRC Press, 2009.

- [33] Y. He, "Rapid Thermal Conductivity Measurement with a Hot Disk Sensor: Part 1. Theoretical Considerations," *Thermochimica Acta*, vol. 436, no. 1-2, pp. 122-129, 01 October 2005.
- [34] S. Gustafsson, "Device for Measuring Thermal Properties of a Test Substance-the Transient Plane Source (TPS) Method". United States of America Patent US5044767 A, 03 September 1991.
- [35] D. G. Gilmore and M. Donabedian, Eds., Spacecraft Thermal Control Handbook:Cryogenics, vol. 02, AIAA, 2003.
- [36] K. K. Chawla, "Micromechanics of Composites," in *Composite Materials*, New York, New York: Springer, 2012, pp. 337-385.
- [37] V. Bohac, M. K. Gustavsson, L. Kubicar and S. E. Gustafsson, "Parameter Estimations for Measurements of Thermal Transport Properties with the Hot Disk Thermal Constant Analyzer," *Review of Scientific Instruments*, vol. 71, no. 6, pp. 2452-2455, June 2000.
- [38] S. Barsi, M. Kassemi, C. H. Panzarella and J. I. D. Alexander, "A Tank Self-Pressurization Experiment using a Model Fluid in Normal Gravit," Reno, 2005.
- [39] ThermTest Inc., "History.1-Transient Plane Source Method- Part1," 15 June 2015. [Online]. Available: https://thermtest.com/history-1-transient-plane-source-methodpart-1/.
- [40] Truefoam Limited, "Expanded vs. Extruded Polystyrene," 2016. [Online]. Available: http://www.truefoam.com/about-eps/expanded-vs-extruded-polystyrene.
- [41] I. M. Ward and J. Sweeney, An Introduction to the Mechanical Properties of Solid Polymers, 2nd ed., Chichester: John Wiley & Sons Ltd., 2004.
- [42] W. A. Campbell, Jr., R. S. Marriott and J. J. Park, "Outgassing Data for Selecting Spacecraft Materials," Greenbelt, 1984.
- [43] S. A. Al-Ajlan, "Measurements of Thermal Properties of Insulation Materials by Using Transient Plane Source Technique," *Applied Thermal engineering*, vol. 26, no. 17-18, pp. 2184-2191, December 2006.
- <sup>[44]</sup> "Variations of Thermal Conductivity of Insulation Materials," *Journal of Architectural Engineering*, vol. 8, no. 4, pp. 125-132, December 2002.

APPENDICES

## APPENDIX A

# Measurement results at 313K

|                 |                     |                  |         |                         |                        | SS 316                      |                  |                   |                        |                        |                   |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |
| 24              | 80                  | 9.868            | 75-150  | 13.916                  | 3.510                  | 3.964                       | 16.8             | 0.264             | 1.21                   | 0.721                  | 4.47E-05          |
| 24              | 80                  | 9.868            | 75-151  | 14.109                  | 3.611                  | 3.908                       | 17.0             | 0.624             | 3.73                   | 0.742                  | 1.03E-04          |
| 24              | 80                  | 9.868            | 75-152  | 14.102                  | 3.593                  | 3.925                       | 17.0             | 0.624             | 3.73                   | 0.738                  | 1.05E-04          |
| 24              | 80                  | 9.868            | 75-153  | 14.129                  | 3.591                  | 3.935                       | 16.9             | 0.623             | 3.72                   | 0.737                  | 8.12E-05          |
| 24              | 80                  | 9.868            | 75-154  | 14.108                  | 3.583                  | 3.937                       | 16.9             | 0.624             | 3.72                   | 0.736                  | 8.20E-05          |
| 24              | 80                  | 9.868            | 75-155  | 14.148                  | 3.612                  | 3.917                       | 17.0             | 0.622             | 3.72                   | 0.742                  | 1.58E-04          |
| 24              | 80                  | 9.868            | 75-156  | 14.114                  | 3.606                  | 3.914                       | 17.0             | 0.624             | 3.73                   | 0.741                  | 1.68E-04          |
| 24              | 80                  | 9.868            | 75-157  | 14.098                  | 3.604                  | 3.912                       | 17.0             | 0.624             | 3.74                   | 0.740                  | 1.70E-04          |
| 24              | 80                  | 9.868            | 75-158  | 14.115                  | 3.614                  | 3.905                       | 17.0             | 0.623             | 3.73                   | 0.742                  | 1.59E-04          |
| 24              | 80                  | 9.868            | 75-159  | 14.102                  | 3.588                  | 3.930                       | 16.9             | 0.624             | 3.75                   | 0.737                  | 9.77E-05          |
| 24              | 80                  | 9.868            | 75-160  | 14.113                  | 3.596                  | 3.925                       | 17.0             | 0.624             | 3.72                   | 0.738                  | 9.00E-05          |
| 24              | 80                  | 9.868            | 75-161  | 14.112                  | 3.612                  | 3.907                       | 17.0             | 0.623             | 3.73                   | 0.742                  | 1.66E-04          |
| 24              | 80                  | 9.868            | 75-162  | 14.137                  | 3.612                  | 3.914                       | 17.0             | 0.623             | 3.73                   | 0.742                  | 1.61E-04          |
| 24              | 80                  | 9.868            | 75-163  | 14.113                  | 3.587                  | 3.934                       | 16.9             | 0.623             | 3.74                   | 0.737                  | 7.67E-05          |
| -               | _                   | -                | Mean    | 14.101                  | 3.594                  | 3.923                       | 17.0             | 0.598             | 3.55                   | 0.738                  | 1.19E-04          |
| -               | _                   | -                | St. Dev | 0.055                   | 0.026                  | 0.016                       | 0.1              | 0.096             | 0.67                   | 0.005                  | 4.29E-05          |

|                 | Polystyrene         |                  |         |                         |                        |                             |                  |                   |                        |                        |                   |  |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |  |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |
| 24              | 80                  | 9.868            | 75-150  | 0.032                   | 0.763                  | 0.042                       | 13.5             | 1.068             | 5.19                   | 0.470                  | 1.50E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.032                   | 0.771                  | 0.041                       | 13.6             | 1.070             | 5.20                   | 0.475                  | 2.03E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.032                   | 0.769                  | 0.041                       | 13.6             | 1.069             | 5.20                   | 0.474                  | 2.11E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.031                   | 0.760                  | 0.041                       | 13.5             | 1.072             | 5.18                   | 0.468                  | 1.54E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.031                   | 0.761                  | 0.041                       | 13.5             | 1.073             | 5.18                   | 0.469                  | 1.95E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.032                   | 0.774                  | 0.041                       | 13.6             | 1.069             | 5.18                   | 0.477                  | 2.02E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.032                   | 0.764                  | 0.041                       | 13.5             | 1.070             | 5.19                   | 0.471                  | 1.33E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.031                   | 0.769                  | 0.041                       | 13.6             | 1.073             | 5.21                   | 0.474                  | 2.00E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.031                   | 0.759                  | 0.041                       | 13.5             | 1.072             | 5.21                   | 0.468                  | 2.17E-04          |  |
| 24              | 80                  | 9.868            | 75-150  | 0.032                   | 0.763                  | 0.041                       | 13.5             | 1.072             | 5.18                   | 0.470                  | 1.42E-04          |  |
| _               | _                   | -                | Mean    | 0.032                   | 0.765                  | 0.041                       | 13.6             | 1.071             | 5.19                   | 0.472                  | 1.81E-04          |  |
| -               | _                   | -                | St. Dev | 7.12E-05                | 4.93E-03               | 2.61E-04                    | 4.37E-02         | 1.85E-03          | 1.31E-02               | 3.04E-03               | 3.19E-05          |  |

|                 | RTV-655             |                  |         |                         |                        |                             |                  |                   |                        |                        |                   |  |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |  |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.103                  | 1.580                       | 11.486           | 1.401             | 4.330                  | 0.803                  | 1.14E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.105                  | 1.555                       | 11.577           | 1.397             | 4.323                  | 0.816                  | 3.59E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.104                  | 1.572                       | 11.514           | 1.398             | 4.322                  | 0.807                  | 1.37E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.104                  | 1.573                       | 11.518           | 1.398             | 4.334                  | 0.808                  | 1.41E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.104                  | 1.572                       | 11.514           | 1.400             | 4.325                  | 0.807                  | 1.18E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.103                  | 1.573                       | 11.509           | 1.399             | 4.324                  | 0.807                  | 1.56E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.103                  | 1.577                       | 11.502           | 1.399             | 4.309                  | 0.806                  | 1.30E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.104                  | 1.569                       | 11.519           | 1.398             | 4.332                  | 0.808                  | 1.24E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.103                  | 1.577                       | 11.505           | 1.398             | 4.333                  | 0.806                  | 1.51E-04          |  |
| 50              | 320                 | 6.403            | 50-200  | 0.163                   | 0.104                  | 1.565                       | 11.531           | 1.401             | 4.336                  | 0.810                  | 1.34E-04          |  |
| _               | _                   | _                | Mean    | 0.163                   | 0.104                  | 1.572                       | 11.5             | 1.399             | 4.33                   | 0.808                  | 1.56E-04          |  |
| -               | _                   | -                | St. Dev | 1.58E-04                | 4.34E-04               | 7.20E-03                    | 2.41E-02         | 1.31E-03          | 7.96E-03               | 3.38E-03               | 7.23E-05          |  |

|                 | VR17                |                  |         |                         |                        |                             |                  |                   |                        |                        |                   |  |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |  |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |
| 35              | 640                 | 6.403            | 50-180  | 0.131                   | 0.066                  | 1.982                       | 11.987           | 1.061             | 4.339                  | 0.875                  | 1.15E-03          |  |
| 35              | 640                 | 6.403            | 50-170  | 0.131                   | 0.066                  | 1.978                       | 12.000           | 1.060             | 4.336                  | 0.877                  | 1.18E-03          |  |
| 35              | 640                 | 6.403            | 50-169  | 0.131                   | 0.067                  | 1.953                       | 12.033           | 1.056             | 4.365                  | 0.882                  | 1.64E-03          |  |
| 35              | 640                 | 6.403            | 50-171  | 0.131                   | 0.066                  | 1.990                       | 11.996           | 1.066             | 4.348                  | 0.876                  | 1.14E-03          |  |
| 35              | 640                 | 6.403            | 50-168  | 0.131                   | 0.067                  | 1.955                       | 12.001           | 1.048             | 4.334                  | 0.877                  | 1.35E-03          |  |
| 35              | 640                 | 6.403            | 50-168  | 0.131                   | 0.067                  | 1.960                       | 12.003           | 1.048             | 4.339                  | 0.877                  | 1.16E-03          |  |
| 35              | 640                 | 6.403            | 50-172  | 0.131                   | 0.065                  | 2.001                       | 12.000           | 1.071             | 4.340                  | 0.877                  | 1.35E-03          |  |
| 35              | 640                 | 6.403            | 50-166  | 0.131                   | 0.068                  | 1.931                       | 12.006           | 1.040             | 4.343                  | 0.878                  | 1.14E-03          |  |
| 35              | 640                 | 6.403            | 50-172  | 0.131                   | 0.065                  | 1.999                       | 12.007           | 1.071             | 4.348                  | 0.878                  | 1.22E-03          |  |
| 35              | 640                 | 6.403            | 50-173  | 0.130                   | 0.065                  | 2.001                       | 12.008           | 1.080             | 4.367                  | 0.878                  | 1.30E-03          |  |
| -               | -                   | _                | Mean    | 0.131                   | 0.066                  | 1.975                       | 12.0             | 1.060             | 4.35                   | 0.878                  | 1.26E-03          |  |
| -               | _                   | -                | St. Dev | 2.39E-04                | 8.81E-04               | 2.43E-02                    | 1.20E-02         | 1.25E-02          | 1.15E-02               | 1.76E-03               | 1.56E-04          |  |

|                 | VR34                |                  |         |                         |                        |                             |                  |                   |                        |                        |                   |  |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |  |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |
| 25              | 640                 | 6.403            | 50-145  | 0.083                   | 0.078                  | 1.065                       | 12.0             | 1.041             | 3.78                   | 0.879                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-144  | 0.082                   | 0.079                  | 1.048                       | 12.0             | 1.039             | 3.84                   | 0.881                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-147  | 0.083                   | 0.076                  | 1.086                       | 12.0             | 1.058             | 3.83                   | 0.872                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-146  | 0.083                   | 0.077                  | 1.073                       | 12.0             | 1.050             | 3.82                   | 0.877                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-146  | 0.083                   | 0.077                  | 1.072                       | 12.0             | 1.052             | 3.83                   | 0.877                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-147  | 0.083                   | 0.077                  | 1.079                       | 12.0             | 1.056             | 3.82                   | 0.879                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-146  | 0.083                   | 0.078                  | 1.068                       | 12.0             | 1.048             | 3.82                   | 0.882                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-145  | 0.082                   | 0.078                  | 1.052                       | 12.0             | 1.047             | 3.84                   | 0.883                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-147  | 0.083                   | 0.076                  | 1.084                       | 12.0             | 1.058             | 3.83                   | 0.873                  | 0.001             |  |
| 25              | 640                 | 6.403            | 50-147  | 0.083                   | 0.076                  | 1.082                       | 12.0             | 1.057             | 3.82                   | 0.876                  | 0.001             |  |
| _               | -                   | _                | Mean    | 0.083                   | 0.077                  | 1.071                       | 12.0             | 1.051             | 3.82                   | 0.878                  | 1.18E-03          |  |
| -               | _                   | -                | St. Dev | 2.04E-04                | 8.28E-04               | 1.29E-02                    | 2.57E-02         | 6.82E-03          | 1.58E-02               | 3.75E-03               | 4.76E-05          |  |

|                 | VR51                |                  |         |                         |                        |                             |                  |                   |                        |                        |                   |  |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |  |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |
| 25              | 320                 | 6.403            | 100-165 | 0.054                   | 0.137                  | 0.395                       | 12.028           | 0.723             | 4.700                  | 0.881                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-166 | 0.054                   | 0.136                  | 0.396                       | 12.041           | 0.732             | 4.704                  | 0.883                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-164 | 0.054                   | 0.137                  | 0.394                       | 11.996           | 0.715             | 4.692                  | 0.876                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-165 | 0.054                   | 0.137                  | 0.394                       | 12.019           | 0.725             | 4.727                  | 0.880                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-163 | 0.054                   | 0.139                  | 0.386                       | 12.045           | 0.709             | 4.704                  | 0.884                  | 0.001             |  |
| 25              | 320                 | 6.403            | 100-163 | 0.054                   | 0.139                  | 0.389                       | 12.031           | 0.708             | 4.710                  | 0.882                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-166 | 0.054                   | 0.135                  | 0.402                       | 11.970           | 0.732             | 4.716                  | 0.873                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-172 | 0.054                   | 0.131                  | 0.415                       | 12.006           | 0.781             | 4.715                  | 0.878                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-165 | 0.054                   | 0.137                  | 0.394                       | 12.034           | 0.723             | 4.723                  | 0.882                  | 0.000             |  |
| 25              | 320                 | 6.403            | 100-164 | 0.054                   | 0.137                  | 0.394                       | 12.001           | 0.715             | 4.714                  | 0.877                  | 0.000             |  |
| _               | _                   | _                | Mean    | 0.054                   | 0.137                  | 0.396                       | 12.017           | 0.726             | 4.710                  | 0.879                  | 2.54E-04          |  |
| -               | _                   | -                | St. Dev | 1.72E-04                | 2.28E-03               | 7.92E-03                    | 2.36E-02         | 2.09E-02          | 1.05E-02               | 3.45E-03               | 1.39E-04          |  |

|                 | PI                  |                  |         |                         |                        |                             |                  |                   |                        |                        |                   |  |
|-----------------|---------------------|------------------|---------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points  | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to Char.<br>Time | Mean<br>Deviation |  |
| [mW]            | [s]                 | [mm]             |         | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.323                  | 0.140                       | 3.592            | 0.907             | 4.280                  | 0.278                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.334                  | 0.136                       | 3.653            | 0.906             | 4.284                  | 0.287                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.340                  | 0.133                       | 3.686            | 0.909             | 4.286                  | 0.292                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.328                  | 0.138                       | 3.623            | 0.908             | 4.299                  | 0.283                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.332                  | 0.136                       | 3.644            | 0.910             | 4.283                  | 0.286                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.327                  | 0.138                       | 3.618            | 0.909             | 4.272                  | 0.282                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.328                  | 0.138                       | 3.624            | 0.908             | 4.269                  | 0.283                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.325                  | 0.139                       | 3.603            | 0.909             | 4.265                  | 0.279                  | 0.000             |  |
| 7               | 80                  | 3.189            | 10-25   | 0.045                   | 0.335                  | 0.135                       | 3.662            | 0.910             | 4.259                  | 0.289                  | 0.000             |  |
| -               | _                   | -                | Mean    | 0.045                   | 0.330                  | 0.137                       | 3.634            | 0.908             | 4.278                  | 0.284                  | 0.000             |  |
| _               | -                   | _                | St. Dev | 7.32E-05                | 5.40E-03               | 2.07E-03                    | 2.97E-02         | 1.15E-03          | 1.24E-02               | 4.65E-03               | 6.55E-05          |  |

| Polystyrene    |               |           |                 |             |  |  |  |  |  |
|----------------|---------------|-----------|-----------------|-------------|--|--|--|--|--|
| Times          | Secole Hast   | Reference | Comula Increase | Total Temp. |  |  |  |  |  |
| Time           | Specific Heat | Increase  | Sample Increase | Increase    |  |  |  |  |  |
| [s]            | [Jkg-1K-1]    | [K]       | [K]             | [K]         |  |  |  |  |  |
| 160            | 977           | 2.60      | 2.60            | 6.43        |  |  |  |  |  |
| 160            | 971           | 2.60      | 2.61            | 6.48        |  |  |  |  |  |
| 160            | 930           | 2.60      | 2.61            | 6.45        |  |  |  |  |  |
| 160            | 1123          | 2.60      | 2.61            | 6.46        |  |  |  |  |  |
| 160            | 1094          | 2.60      | 2.61            | 6.49        |  |  |  |  |  |
| 160            | 1046          | 2.60      | 2.62            | 6.47        |  |  |  |  |  |
| 160            | 1066          | 2.60      | 2.62            | 6.49        |  |  |  |  |  |
| 160            | 896           | 2.60      | 2.65            | 6.56        |  |  |  |  |  |
| 160            | 1220          | 2.60      | 2.60            | 6.44        |  |  |  |  |  |
| 160            | 1414          | 2.60      | 2.60            | 6.50        |  |  |  |  |  |
| 160            | 1337          | 2.60      | 2.60            | 6.49        |  |  |  |  |  |
| 160            | 1399          | 2.60      | 2.60            | 6.50        |  |  |  |  |  |
| 160            | 1267          | 2.60      | 2.60            | 6.51        |  |  |  |  |  |
| 160            | 1316          | 2.60      | 2.60            | 6.52        |  |  |  |  |  |
| 160            | 1355          | 2.60      | 2.60            | 6.51        |  |  |  |  |  |
| 160            | 1350          | 2.60      | 2.60            | 6.50        |  |  |  |  |  |
| 160            | 950           | 2.60      | 2.60            | 6.45        |  |  |  |  |  |
| 160            | 939           | 2.60      | 2.60            | 6.46        |  |  |  |  |  |
| 160            | 824           | 2.60      | 2.60            | 6.45        |  |  |  |  |  |
| 160            | 1147          | 2.60      | 2.60            | 6.45        |  |  |  |  |  |
| 160 [Average]  | 1131          | 3         | 3               | 6           |  |  |  |  |  |
| 160 [Std. Dev] | 188.45        | 0.00      | 0.01            | 0.03        |  |  |  |  |  |

| RTV-655 |                  |                       |                    |                            |  |  |  |  |  |  |
|---------|------------------|-----------------------|--------------------|----------------------------|--|--|--|--|--|--|
| Time    | Specific<br>Heat | Reference<br>Increase | Sample<br>Increase | Total<br>Temp.<br>Increase |  |  |  |  |  |  |
| [s]     | [Jkg-1K-<br>1]   | [K]                   | [K]                | [K]                        |  |  |  |  |  |  |
| 160     | 1533             | 2.60                  | 2.60               | 7.24                       |  |  |  |  |  |  |
| 160     | 1554             | 2.60                  | 2.59               | 7.24                       |  |  |  |  |  |  |
| 160     | 1537             | 2.60                  | 2.60               | 7.25                       |  |  |  |  |  |  |
| 160     | 1547             | 2.60                  | 2.60               | 7.23                       |  |  |  |  |  |  |
| 160     | 1543             | 2.60                  | 2.60               | 7.26                       |  |  |  |  |  |  |
| 160     | 1540             | 2.60                  | 2.60               | 7.25                       |  |  |  |  |  |  |
| 160     | 1544             | 2.60                  | 2.60               | 7.24                       |  |  |  |  |  |  |
| 160     | 1542             | 2.60                  | 2.60               | 7.23                       |  |  |  |  |  |  |
| 160     | 1552             | 2.60                  | 2.59               | 7.23                       |  |  |  |  |  |  |
| 160     | 1544             | 2.60                  | 2.60               | 7.24                       |  |  |  |  |  |  |
| 320     | 1605             | 2.21                  | 2.19               | 5.76                       |  |  |  |  |  |  |
| 320     | 1600             | 2.21                  | 2.19               | 5.75                       |  |  |  |  |  |  |
| 320     | 1602             | 2.21                  | 2.19               | 5.75                       |  |  |  |  |  |  |
| 320     | 1606             | 2.21                  | 2.19               | 5.76                       |  |  |  |  |  |  |
| 320     | 1602             | 2.21                  | 2.19               | 5.77                       |  |  |  |  |  |  |
| 320     | 1608             | 2.21                  | 2.19               | 5.77                       |  |  |  |  |  |  |
| 320     | 1611             | 2.21                  | 2.19               | 5.75                       |  |  |  |  |  |  |
| 320     | 1611             | 2.21                  | 2.19               | 5.73                       |  |  |  |  |  |  |
| 320     | 1616             | 2.21                  | 2.18               | 5.74                       |  |  |  |  |  |  |
| 320     | 1605             | 2.21                  | 2.19               | 5.74                       |  |  |  |  |  |  |

| 160 [Average]  | 1544 | 2.60     | 2.60     | 7.24     |
|----------------|------|----------|----------|----------|
| 160 [Std. Dev] | 6.43 | 4.68E-16 | 2.67E-03 | 1.15E-02 |
| 320 [Average]  | 1607 | 2.21     | 2.19     | 5.75     |
| 320 [Std. Dev] | 4.81 | 0.00E+00 | 1.12E-03 | 1.42E-02 |

| VR17 |                  |                       |                    |                            |  |  |  |  |  |  |
|------|------------------|-----------------------|--------------------|----------------------------|--|--|--|--|--|--|
| Time | Specific<br>Heat | Reference<br>Increase | Sample<br>Increase | Total<br>Temp.<br>Increase |  |  |  |  |  |  |
| [s]  | [Jkg-<br>1K-1]   | [K]                   | [K]                | [K]                        |  |  |  |  |  |  |
| 160  | 1258             | 2.60                  | 2.65               | 7.63                       |  |  |  |  |  |  |
| 160  | 1278             | 2.60                  | 2.64               | 7.61                       |  |  |  |  |  |  |
| 160  | 1276             | 2.60                  | 2.64               | 7.61                       |  |  |  |  |  |  |
| 160  | 1274             | 2.60                  | 2.64               | 7.60                       |  |  |  |  |  |  |
| 160  | 1281             | 2.60                  | 2.63               | 7.57                       |  |  |  |  |  |  |
| 160  | 1271             | 2.60                  | 2.64               | 7.61                       |  |  |  |  |  |  |
| 160  | 1277             | 2.60                  | 2.63               | 7.58                       |  |  |  |  |  |  |
| 160  | 1272             | 2.60                  | 2.64               | 7.63                       |  |  |  |  |  |  |
| 160  | 1284             | 2.60                  | 2.63               | 7.60                       |  |  |  |  |  |  |
| 160  | 1272             | 2.60                  | 2.64               | 7.59                       |  |  |  |  |  |  |
| 320  | 1595             | 2.21                  | 2.21               | 6.20                       |  |  |  |  |  |  |
| 320  | 1632             | 2.21                  | 2.19               | 6.15                       |  |  |  |  |  |  |
| 320  | 1631             | 2.21                  | 2.19               | 6.17                       |  |  |  |  |  |  |

| 320            | 1632  | 2.21     | 2.19     | 6.17     |
|----------------|-------|----------|----------|----------|
| 320            | 1631  | 2.21     | 2.19     | 6.15     |
| 320            | 1635  | 2.21     | 2.19     | 6.15     |
| 320            | 1640  | 2.21     | 2.19     | 6.18     |
| 320            | 1636  | 2.21     | 2.19     | 6.16     |
| 320            | 1635  | 2.21     | 2.19     | 6.16     |
| 320            | 1632  | 2.21     | 2.19     | 6.15     |
| 160 [Average]  | 1274  | 2.60     | 2.64     | 7.60     |
| 160 [Std. Dev] | 7.09  | 4.68E-16 | 5.36E-03 | 1.86E-02 |
| 320 [Average]  | 1630  | 2.21     | 2.19     | 6.17     |
| 320 [Std. Dev] | 12.54 | 0.00E+00 | 6.46E-03 | 1.62E-02 |

|      |                  | VR34                  |      |                            |
|------|------------------|-----------------------|------|----------------------------|
| Time | Specific<br>Heat | Reference<br>Increase | VR17 | Total<br>Temp.<br>Increase |
| [8]  | [Jkg-<br>1K-1]   | [K]                   | [K]  | [K]                        |
| 160  | 1241             | 2.60                  | 2.60 | 7.34                       |
| 160  | 1247             | 2.60                  | 2.60 | 7.34                       |
| 160  | 1249             | 2.60                  | 2.60 | 7.36                       |
| 160  | 1248             | 2.60                  | 2.60 | 7.35                       |
| 160  | 1252             | 2.60                  | 2.60 | 7.35                       |
| 160  | 1243             | 2.60                  | 2.60 | 7.35                       |
| 160  | 1245             | 2.60                  | 2.60 | 7.35                       |
| 160  | 1252             | 2.60                  | 2.60 | 7.35                       |

| 160            | 1251 | 2.60     | 2.60     | 7.34     |
|----------------|------|----------|----------|----------|
| 160            | 1243 | 2.60     | 2.60     | 7.36     |
| 320            | 1562 | 2.21     | 2.24     | 6.22     |
| 320            | 1563 | 2.21     | 2.24     | 6.22     |
| 320            | 1567 | 2.21     | 2.24     | 6.21     |
| 320            | 1575 | 2.21     | 2.24     | 6.20     |
| 320            | 1571 | 2.21     | 2.24     | 6.20     |
| 320            | 1566 | 2.21     | 2.24     | 6.21     |
| 320            | 1574 | 2.21     | 2.24     | 6.20     |
| 320            | 1567 | 2.21     | 2.24     | 6.22     |
| 320            | 1560 | 2.21     | 2.24     | 6.23     |
| 320            | 1568 | 2.21     | 2.24     | 6.21     |
| 160 [Average]  | 1247 | 2.60     | 2.60     | 7.35     |
| 160 [Std. Dev] | 3.87 | 4.68E-16 | 1.14E-03 | 6.02E-03 |
| 320 [Average]  | 1567 | 2.21     | 2.24     | 6.21     |
| 320 [Std. Dev] | 4.94 | 0.00E+00 | 1.74E-03 | 1.01E-02 |

| VR51 |                  |                       |                    |                            |  |  |  |  |  |  |  |  |
|------|------------------|-----------------------|--------------------|----------------------------|--|--|--|--|--|--|--|--|
| Time | Specific<br>Heat | Reference<br>Increase | Sample<br>Increase | Total<br>Temp.<br>Increase |  |  |  |  |  |  |  |  |
| [s]  | [Jkg-<br>1K-1]   | [K]                   | [K]                | [K]                        |  |  |  |  |  |  |  |  |
| 160  | 1196             | 2.60                  | 2.63               | 7.36                       |  |  |  |  |  |  |  |  |

| 160            | 1211  | 2.60     | 2.62     | 7.33     |
|----------------|-------|----------|----------|----------|
| 160            | 1210  | 2.60     | 2.62     | 7.35     |
| 160            | 1216  | 2.60     | 2.62     | 7.32     |
| 160            | 1217  | 2.60     | 2.62     | 7.31     |
| 160            | 1219  | 2.60     | 2.62     | 7.32     |
| 160            | 1215  | 2.60     | 2.62     | 7.35     |
| 160            | 1215  | 2.60     | 2.62     | 7.33     |
| 160            | 1222  | 2.60     | 2.62     | 7.32     |
| 160            | 1214  | 2.60     | 2.62     | 7.33     |
| 320            | 1552  | 2.21     | 2.26     | 6.28     |
| 320            | 1584  | 2.21     | 2.25     | 6.26     |
| 320            | 1589  | 2.21     | 2.25     | 6.25     |
| 320            | 1589  | 2.21     | 2.25     | 6.25     |
| 320            | 1590  | 2.21     | 2.25     | 6.23     |
| 320            | 1578  | 2.21     | 2.25     | 6.25     |
| 320            | 1590  | 2.21     | 2.25     | 6.26     |
| 320            | 1595  | 2.21     | 2.25     | 6.24     |
| 320            | 1590  | 2.21     | 2.25     | 6.24     |
| 320            | 1586  | 2.21     | 2.25     | 6.23     |
| 160 [Average]  | 1213  | 2.28     | 2.33     | 6.47     |
| 160 [Std. Dev] | 7.00  | 1.66E-01 | 1.54E-01 | 4.54E-01 |
| 320 [Average]  | 1584  | 2.21     | 2.25     | 6.25     |
| 320 [Std. Dev] | 12.29 | 0.00E+00 | 4.60E-03 | 1.45E-02 |

**PI-Sheets** 

| Time | Specific<br>Heat | Reference<br>Increase | Sample<br>Increase | Total<br>Temp.<br>Increase |
|------|------------------|-----------------------|--------------------|----------------------------|
| [s]  | [Jkg-<br>1K-1]   | [K]                   | [K]                | [K]                        |
| 80   | 1255             | 2.56                  | 2.44               | 6.57                       |
| 80   | 1239             | 2.56                  | 2.45               | 6.60                       |
| 80   | 1230             | 2.56                  | 2.45               | 6.59                       |
| 80   | 1241             | 2.56                  | 2.24               | 6.06                       |
| 80   | 1255             | 2.56                  | 2.44               | 6.57                       |
| 80   | 1239             | 2.56                  | 2.45               | 6.60                       |
| 80   | 1230             | 2.56                  | 2.45               | 6.59                       |
| 80   | 1241             | 2.56                  | 2.24               | 6.06                       |
| 80   | 1268             | 2.56                  | 2.49               | 6.68                       |
| 80   | 1222             | 2.56                  | 2.56               | 6.91                       |
| 80   | 1218             | 2.56                  | 2.60               | 7.00                       |
| 80   | 1255             | 2.56                  | 2.44               | 6.57                       |
| 80   | 1239             | 2.56                  | 2.45               | 6.60                       |
| 80   | 1230             | 2.56                  | 2.45               | 6.59                       |
| 80   | 1241             | 2.56                  | 2.24               | 6.06                       |
| 80   | 1268             | 2.56                  | 2.49               | 6.68                       |
| 80   | 1222             | 2.56                  | 2.56               | 6.91                       |
| 80   | 1218             | 2.56                  | 2.60               | 7.00                       |
| 80   | 1177             | 2.56                  | 2.57               | 6.90                       |
| 80   | 1229             | 2.56                  | 2.56               | 6.88                       |
| 80   | 1234             | 2.56                  | 2.56               | 6.89                       |
| 80   | 1209             | 2.56                  | 2.57               | 6.92                       |

| 80  | 1205 | 2.56 | 2.57 | 6.89 |
|-----|------|------|------|------|
| 160 | 1304 | 2.59 | 2.49 | 6.23 |
| 160 | 1310 | 2.59 | 2.50 | 6.25 |
| 160 | 1286 | 2.59 | 2.52 | 6.35 |
| 160 | 1246 | 2.59 | 2.54 | 6.38 |
| 160 | 1275 | 2.59 | 2.59 | 6.49 |
| 160 | 1263 | 2.59 | 2.64 | 6.62 |
| 160 | 1279 | 2.59 | 2.59 | 6.49 |
| 160 | 1266 | 2.59 | 2.59 | 6.47 |
| 160 | 1305 | 2.59 | 2.59 | 6.48 |
| 160 | 1301 | 2.59 | 2.59 | 6.47 |
| 160 | 1300 | 2.59 | 2.59 | 6.47 |
| 320 | 1253 | 2.38 | 2.31 | 5.91 |
| 320 | 1241 | 2.38 | 2.32 | 5.91 |
| 320 | 1285 | 2.38 | 2.35 | 5.99 |
| 320 | 1253 | 2.38 | 2.31 | 5.91 |
| 320 | 1241 | 2.38 | 2.32 | 5.91 |
| 320 | 1285 | 2.38 | 2.35 | 5.99 |
| 320 | 1218 | 2.38 | 2.37 | 6.05 |
| 320 | 1258 | 2.38 | 2.40 | 6.13 |
| 320 | 1260 | 2.38 | 2.44 | 6.23 |
| 320 | 1253 | 2.38 | 2.31 | 5.91 |
| 320 | 1241 | 2.38 | 2.32 | 5.91 |
| 320 | 1285 | 2.38 | 2.35 | 5.99 |
| 320 | 1218 | 2.38 | 2.37 | 6.05 |
| 320 | 1258 | 2.38 | 2.40 | 6.13 |

| 1              | 1     | 1        | 1        | 1        |
|----------------|-------|----------|----------|----------|
| 320            | 1260  | 2.38     | 2.44     | 6.23     |
| 320            | 1265  | 2.38     | 2.38     | 6.07     |
| 320            | 1326  | 2.38     | 2.38     | 6.06     |
| 320            | 1298  | 2.38     | 2.38     | 6.08     |
| 320            | 1288  | 2.38     | 2.38     | 6.09     |
| 320            | 1252  | 2.38     | 2.38     | 6.08     |
| 80 [Average]   | 1233  | 2.56     | 2.47     | 6.66     |
| 80 [Std. Dev]  | 20.76 | 9.08E-16 | 1.08E-01 | 2.85E-01 |
| 160 [Average]  | 1285  | 2.59     | 2.57     | 6.43     |
| 160 [Std. Dev] | 20.86 | 4.66E-16 | 4.60E-02 | 1.16E-01 |
| 320 [Average]  | 1262  | 2.38     | 2.36     | 6.03     |
| 320 [Std. Dev] | 26.47 | 4.56E-16 | 3.84E-02 | 1.03E-01 |

## APPENDIX A

# MEASUREMENT RESULTS AT 295K

|                 | SS 316              |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |  |  |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.494                  | 3.065                  | 4.403                       | 9.9              | 0.847             | 5.12                   | 0.252                  | 3.51E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.489                  | 3.066                  | 4.399                       | 9.9              | 0.847             | 5.06                   | 0.252                  | 3.72E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.452                  | 3.065                  | 4.388                       | 9.9              | 0.849             | 5.04                   | 0.252                  | 3.64E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.462                  | 3.053                  | 4.409                       | 9.9              | 0.848             | 5.06                   | 0.251                  | 3.63E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.458                  | 3.049                  | 4.414                       | 9.9              | 0.847             | 5.09                   | 0.250                  | 3.70E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.460                  | 3.057                  | 4.403                       | 9.9              | 0.847             | 5.04                   | 0.251                  | 3.62E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.491                  | 3.058                  | 4.412                       | 9.9              | 0.846             | 5.07                   | 0.251                  | 3.62E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.468                  | 3.057                  | 4.406                       | 9.9              | 0.848             | 5.10                   | 0.251                  | 3.53E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.548                  | 3.073                  | 4.409                       | 9.9              | 0.844             | 4.98                   | 0.252                  | 3.65E-04          |  |  |
| 3500            | 20                  | 9.868            | 15-80     | 13.498                  | 3.062                  | 4.408                       | 9.9              | 0.846             | 5.07                   | 0.252                  | 3.65E-04          |  |  |
| -               | -                   | -                | Mean      | 13.482                  | 3.060                  | 4.405                       | 9.9              | 0.847             | 5.06                   | 0.251                  | 3.63E-04          |  |  |
| -               | -                   | -                | Std. Dev. | 0.029                   | 0.007                  | 0.007                       | 0.0              | 0.001             | 0.04                   | 0.001                  | 6.59E-06          |  |  |

|                 | Polystyrene         |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |  |  |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.702                  | 0.042                       | 8.2              | 0.877             | 2.72                   | -                      | 3.79E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.699                  | 0.042                       | 8.2              | 0.879             | 2.75                   | -                      | 4.15E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.688                  | 0.042                       | 8.1              | 0.878             | 2.75                   | -                      | 4.74E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.707                  | 0.041                       | 8.2              | 0.880             | 2.76                   | -                      | 4.43E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.708                  | 0.041                       | 8.2              | 0.883             | 2.67                   | -                      | 4.26E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.681                  | 0.043                       | 8.1              | 0.888             | 2.77                   | -                      | 4.03E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.698                  | 0.042                       | 8.2              | 0.885             | 2.70                   | -                      | 4.12E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.699                  | 0.042                       | 8.2              | 0.883             | 2.70                   | -                      | 4.67E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.703                  | 0.041                       | 8.2              | 0.883             | 2.71                   | -                      | 5.66E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    | 0.029                   | 0.704                  | 0.041                       | 8.2              | 0.884             | 2.69                   | -                      | 4.72E-04          |  |  |
| 8               | 40                  | 6.403            | 40-120    |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
| 8               | 40                  | 6.403            | 40-120    |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
| -               | _                   | -                | Mean      | 0.029                   | 0.699                  | 0.042                       | 8.2              | 0.882             | 2.72                   | #DIV/0!                | 4.46E-04          |  |  |
| -               | _                   | _                | Std. Dev. | 1.02E-04                | 8.56E-03               | 4.58E-04                    | 5.04E-02         | 3.42E-03          | 3.18E-02               | #DIV/0!                | 5.29E-05          |  |  |

|                 | RTV-655             |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |  |  |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.109                  | 1.540                       | 11.494           | 1.927             | 3.426                  | 0.805                  | 1.55E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.107                  | 1.551                       | 11.427           | 1.934             | 3.443                  | 0.795                  | 1.58E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.166                   | 0.107                  | 1.549                       | 11.427           | 1.934             | 3.450                  | 0.795                  | 1.52E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.109                  | 1.537                       | 11.500           | 1.928             | 3.434                  | 0.805                  | 1.82E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.108                  | 1.545                       | 11.447           | 1.935             | 3.434                  | 0.798                  | 1.74E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.108                  | 1.545                       | 11.467           | 1.931             | 3.433                  | 0.801                  | 2.19E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.168                   | 0.110                  | 1.528                       | 11.555           | 1.923             | 3.422                  | 0.813                  | 2.22E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.168                   | 0.109                  | 1.539                       | 11.507           | 1.926             | 3.417                  | 0.806                  | 1.82E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.108                  | 1.543                       | 11.472           | 1.931             | 3.424                  | 0.802                  | 1.80E-04          |  |  |
| 40              | 320                 | 6.403            | 15-190    | 0.167                   | 0.109                  | 1.535                       | 11.501           | 1.932             | 3.432                  | 0.806                  | 1.75E-04          |  |  |
| -               | -                   | _                | Mean      | 0.167                   | 0.108                  | 1.541                       | 11.5             | 1.930             | 3.43                   | 0.803                  | 1.80E-04          |  |  |
| -               | _                   | -                | Std. Dev. | 4.57E-04                | 7.55E-04               | 7.01E-03                    | 4.00E-02         | 3.95E-03          | 9.89E-03               | 5.59E-03               | 2.40E-05          |  |  |

|                 | VR17                |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |  |  |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |  |
| 30              | 640                 | 6.403            | 75-138    | 0.120                   | 0.081                  | 1.473                       | 11.995           | 0.483             | 3.371                  | 0.876                  | 2.46E-04          |  |  |
| 30              | 640                 | 6.403            | 75-176    | 0.118                   | 0.063                  | 1.869                       | 11.951           | 0.694             | 3.470                  | 0.870                  | 2.70E-04          |  |  |
| 30              | 640                 | 6.403            | 75-117    | 0.124                   | 0.097                  | 1.286                       | 12.025           | 0.338             | 3.254                  | 0.881                  | 2.17E-04          |  |  |
| 30              | 640                 | 6.403            | 75-135    | 0.124                   | 0.083                  | 1.483                       | 12.003           | 0.452             | 3.332                  | 0.877                  | 2.35E-04          |  |  |
| 30              | 640                 | 6.403            | 75-139    | 0.122                   | 0.081                  | 1.502                       | 12.019           | 0.481             | 3.336                  | 0.880                  | 4.55E-04          |  |  |
| 30              | 640                 | 6.403            | 75-141    | 0.122                   | 0.080                  | 1.529                       | 11.987           | 0.494             | 3.349                  | 0.875                  | 2.43E-04          |  |  |
| 30              | 640                 | 6.403            | 75-146    | 0.122                   | 0.077                  | 1.576                       | 12.010           | 0.523             | 3.372                  | 0.878                  | 2.38E-04          |  |  |
| 30              | 640                 | 6.403            | 75-142    | 0.121                   | 0.080                  | 1.520                       | 12.028           | 0.502             | 3.354                  | 0.881                  | 2.76E-04          |  |  |
| 30              | 640                 | 6.403            | 75-145    | 0.122                   | 0.078                  | 1.574                       | 11.997           | 0.515             | 3.373                  | 0.877                  | 2.49E-04          |  |  |
| 30              | 640                 | 6.403            | 75-142    | 0.123                   | 0.079                  | 1.555                       | 11.972           | 0.497             | 3.309                  | 0.873                  | 2.51E-04          |  |  |
| _               | _                   | _                | Mean      | 0.122                   | 0.080                  | 1.537                       | 12.0             | 0.498             | 3.35                   | 0.877                  | 2.68E-04          |  |  |
| -               | _                   | -                | Std. Dev. | 1.66E-03                | 8.03E-03               | 1.43E-01                    | 2.41E-02         | 8.66E-02          | 5.50E-02               | 3.52E-03               | 6.79E-05          |  |  |

|                 | VR34                |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |  |  |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|--|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |  |  |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.082                   | 0.077                  | 1.061                       | 12.0             | 0.844             | 3.26                   | 0.873                  | 8.04E-04          |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.082                   | 0.078                  | 1.053                       | 12.0             | 0.838             | 3.26                   | 0.882                  | 8.75E-04          |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.083                   | 0.077                  | 1.072                       | 12.0             | 0.835             | 3.25                   | 0.873                  | 7.49E-04          |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.082                   | 0.078                  | 1.060                       | 12.0             | 0.841             | 3.28                   | 0.877                  | 7.45E-04          |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.082                   | 0.078                  | 1.055                       | 12.0             | 0.842             | 3.28                   | 0.877                  | 7.88E-04          |  |  |
| 20              | 640                 | 6.403            | 50-144    | 0.082                   | 0.079                  | 1.040                       | 12.0             | 0.837             | 3.28                   | 0.884                  | 8.20E-04          |  |  |
| 20              | 640                 | 6.403            | 50-144    | 0.083                   | 0.078                  | 1.056                       | 12.0             | 0.830             | 3.26                   | 0.878                  | 8.22E-04          |  |  |
| 20              | 640                 | 6.403            | 50-142    | 0.082                   | 0.079                  | 1.037                       | 12.0             | 0.823             | 3.29                   | 0.876                  | 8.12E-04          |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.082                   | 0.078                  | 1.050                       | 12.0             | 0.839             | 3.29                   | 0.883                  | 8.29E-04          |  |  |
| 20              | 640                 | 6.403            | 50-145    | 0.082                   | 0.077                  | 1.064                       | 12.0             | 0.840             | 3.28                   | 0.874                  | 7.73E-04          |  |  |
| -               | -                   | -                | Mean      | 0.082                   | 0.078                  | 1.055                       | 12.0             | 0.837             | 3.27                   | 0.878                  | 8.02E-04          |  |  |
| -               | -                   | -                | Std. Dev. | 3.11E-04                | 6.54E-04               | 1.05E-02                    | 2.77E-02         | 6.20E-03          | 1.58E-02               | 4.05E-03               | 3.94E-05          |  |  |

| VR51            |                     |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |
| 10              | 40                  | 3.189            | 100-180   | 0.053                   | 0.125                  | 0.422                       | 12.016           | 0.697             | 3.891                  | 0.879                  | 3.06E-04          |
| 10              | 40                  | 3.189            | 100-181   | 0.053                   | 0.125                  | 0.424                       | 12.035           | 0.704             | 3.892                  | 0.882                  | 2.42E-04          |
| 10              | 40                  | 3.189            | 100-175   | 0.053                   | 0.128                  | 0.417                       | 11.957           | 0.661             | 3.872                  | 0.871                  | 2.41E-04          |
| 10              | 40                  | 3.189            | 100-190   | 0.053                   | 0.119                  | 0.444                       | 12.033           | 0.765             | 3.910                  | 0.882                  | 3.01E-04          |
| 10              | 40                  | 3.189            | 100-173   | 0.053                   | 0.130                  | 0.410                       | 12.013           | 0.644             | 3.890                  | 0.879                  | 2.13E-04          |
| 20              | 40                  | 3.189            | 100-166   | 0.054                   | 0.135                  | 0.400                       | 11.969           | 0.590             | 3.840                  | 0.872                  | 1.91E-04          |
| 20              | 40                  | 3.189            | 100-174   | 0.054                   | 0.129                  | 0.414                       | 12.003           | 0.649             | 3.857                  | 0.877                  | 2.83E-04          |
| 20              | 40                  | 3.189            | 100-172   | 0.053                   | 0.131                  | 0.406                       | 12.016           | 0.638             | 3.881                  | 0.879                  | 2.43E-04          |
| 20              | 40                  | 3.189            | 100-169   | 0.054                   | 0.134                  | 0.401                       | 12.018           | 0.613             | 3.850                  | 0.880                  | 2.36E-04          |
| 20              | 40                  | 3.189            | 100-164   | 0.054                   | 0.137                  | 0.390                       | 12.000           | 0.578             | 3.835                  | 0.877                  | 2.05E-04          |
| _               | _                   | -                | Mean      | 0.053                   | 0.129                  | 0.413                       | 12.006           | 0.654             | 3.872                  | 0.878                  | 2.46E-04          |
| -               | _                   | -                | Std. Dev. | 3.60E-04                | 5.34E-03               | 1.50E-02                    | 2.54E-02         | 5.66E-02          | 2.52E-02               | 3.71E-03               | 3.93E-05          |
|                 | PI                  |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |
| 10              | 40                  | 3.189            | 10-25     | 0.050                   | 0.318                  | 0.157                       | 2.520            | 0.967             | 5.125                  | 0.137                  | 1.78E-04          |
| 10              | 40                  | 3.189            | 10-25     | 0.049                   | 0.310                  | 0.158                       | 2.492            | 0.976             | 5.163                  | 0.134                  | 2.05E-04          |
| 10              | 40                  | 3.189            | 10-25     | 0.049                   | 0.303                  | 0.161                       | 2.463            | 0.977             | 5.165                  | 0.131                  | 2.32E-04          |
| 10              | 40                  | 3.189            | 10-25     | 0.049                   | 0.301                  | 0.162                       | 2.454            | 0.978             | 5.171                  | 0.130                  | 1.83E-04          |
| 10              | 40                  | 3.189            | 10-25     | 0.049                   | 0.301                  | 0.161                       | 2.453            | 0.981             | 5.165                  | 0.129                  | 2.45E-04          |
| 20              | 40                  | 3.189            | 10-25     | 0.047                   | 0.274                  | 0.173                       | 2.565            | 0.881             | 4.052                  | 0.142                  | 8.71E-05          |
| 20              | 40                  | 3.189            | 10-25     | 0.047                   | 0.266                  | 0.177                       | 2.526            | 0.879             | 4.062                  | 0.137                  | 3.88E-04          |
| 20              | 40                  | 3.189            | 10-25     | 0.048                   | 0.283                  | 0.170                       | 2.605            | 0.878             | 4.039                  | 0.146                  | 2.96E-04          |
| 20              | 40                  | 3.189            | 10-25     | 0.048                   | 0.284                  | 0.169                       | 2.613            | 0.880             | 4.042                  | 0.147                  | 2.51E-04          |
| 20              | 40                  | 3.189            | 10-25     | 0.048                   | 0.284                  | 0.170                       | 2.610            | 0.875             | 4.042                  | 0.147                  | 1.78E-04          |
| _               | _                   | _                | Mean      | 0.0484                  | 0.292                  | 0.166                       | 2.530            | 0.927             | 4.603                  | 0.138                  | 2.24E-04          |
| -               | _                   | -                | Std. Dev. | 8.03E-04                | 1.66E-02               | 6.87E-03                    | 6.49E-02         | 5.14E-02          | 5.85E-01               | 7.07E-03               | 8.05E-05          |

|                 | Paraffin Wax        |                  |           |                         |                        |                             |                  |                   |                        |                        |                   |
|-----------------|---------------------|------------------|-----------|-------------------------|------------------------|-----------------------------|------------------|-------------------|------------------------|------------------------|-------------------|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points    | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific Heat | Probing<br>Depth | Temp.<br>Increase | Total Temp<br>Increase | Total to<br>Char. Time | Mean<br>Deviation |
| [mW]            | [s]                 | [mm]             |           | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-1]                  | [mm]             | [K]               | [K]                    |                        | [K]               |
| 100             | 160                 | 6.403            | 50-200    | 0.260                   | 0.118                  | 2.209                       | 8.683            | 1.749             | 5.077                  | 0.459                  | 4.17E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.258                   | 0.118                  | 2.184                       | 8.688            | 1.766             | 5.096                  | 0.460                  | 4.68E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.262                   | 0.122                  | 2.155                       | 8.824            | 1.739             | 5.098                  | 0.474                  | 5.04E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.254                   | 0.112                  | 2.279                       | 8.451            | 1.781             | 5.139                  | 0.435                  | 5.45E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.262                   | 0.124                  | 2.111                       | 8.904            | 1.741             | 5.147                  | 0.483                  | 5.66E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.258                   | 0.118                  | 2.194                       | 8.681            | 1.760             | 5.149                  | 0.459                  | 6.66E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.260                   | 0.120                  | 2.168                       | 8.758            | 1.753             | 5.168                  | 0.467                  | 5.44E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.260                   | 0.120                  | 2.168                       | 8.768            | 1.749             | 5.170                  | 0.468                  | 2.34E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.258                   | 0.115                  | 2.238                       | 8.590            | 1.760             | 5.174                  | 0.449                  | 8.05E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.258                   | 0.115                  | 2.246                       | 8.576            | 1.759             | 5.177                  | 0.448                  | 7.59E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.261                   | 0.117                  | 2.223                       | 8.660            | 1.744             | 5.179                  | 0.457                  | 5.01E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.257                   | 0.112                  | 2.294                       | 8.468            | 1.764             | 5.196                  | 0.437                  | 4.60E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.266                   | 0.124                  | 2.141                       | 8.912            | 1.717             | 5.198                  | 0.484                  | 4.69E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.262                   | 0.118                  | 2.229                       | 8.672            | 1.738             | 5.211                  | 0.458                  | 5.30E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.264                   | 0.127                  | 2.086                       | 9.004            | 1.727             | 5.212                  | 0.494                  | 6.23E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.263                   | 0.124                  | 2.129                       | 8.891            | 1.735             | 5.216                  | 0.481                  | 1.86E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.263                   | 0.123                  | 2.132                       | 8.884            | 1.736             | 5.223                  | 0.481                  | 2.23E-04          |
| 100             | 160                 | 6.403            | 50-200    | 0.261                   | 0.123                  | 2.125                       | 8.870            | 1.742             | 5.232                  | 0.479                  | 3.18E-04          |
| -               | -                   | -                | Mean      | 0.260                   | 0.119                  | 2.184                       | 8.738            | 1.748             | 0.465                  | 0.000                  | 18.000            |
| _               | -                   | -                | Std. Dev. | 2.82E-03                | 4.30E-03               | 5.91E-02                    | 1.58E-01         | 1.56E-02          | 1.67E-02               | 1.71E-04               |                   |

| Paraffin Wax    |               |                    |                 |                      |  |  |
|-----------------|---------------|--------------------|-----------------|----------------------|--|--|
| Time            | Specific Heat | Reference Increase | Sample Increase | Total Temp. Increase |  |  |
| [8]             | [Jkg-1K-1]    | [K]                | [K]             | [K]                  |  |  |
| 320             | 2182          | 2.70               | 2.88            | 5.56                 |  |  |
| 640             | 2464          | 3.43               | 3.32            | 4.85                 |  |  |
| 640             | 2417          | 2.73               | 2.79            | 5.16                 |  |  |
| 640             | 2536          | 2.73               | 2.71            | 5.03                 |  |  |
| 640             | 2546          | 2.73               | 2.71            | 5.04                 |  |  |
| 640             | 2544          | 2.73               | 2.71            | 5.04                 |  |  |
| 640             | 2542          | 2.73               | 2.71            | 5.05                 |  |  |
| 640             | 2533          | 2.73               | 2.72            | 5.05                 |  |  |
| 640             | 2523          | 2.73               | 2.72            | 5.06                 |  |  |
| 640             | 2490          | 2.73               | 2.74            | 5.09                 |  |  |
| 640             | 2492          | 2.73               | 2.74            | 5.09                 |  |  |
| 640             | 2456          | 2.73               | 2.76            | 5.10                 |  |  |
| 1280            | 2459          | 3.18               | 3.26            | 4.60                 |  |  |
| 320 [Average]   | 2182          | 3                  | 3               | 6                    |  |  |
| 320 [Std. Dev]  | #DIV/0!       | #DIV/0!            | #DIV/0!         | #DIV/0!              |  |  |
| 640 [Average]   | 2504          | 3                  | 3               | 5                    |  |  |
| 640 [Std. Dev]  | 43            | 0.21               | 0.18            | 0.07                 |  |  |
| 1280 [Average]  | 2459          | 3                  | 3               | 5                    |  |  |
| 1280 [Std. Dev] | #DIV/0!       | #DIV/0!            | #DIV/0!         | #DIV/0!              |  |  |

| Polystyrene (XI | PS) |
|-----------------|-----|
|-----------------|-----|

| Time | Specific       | Reference | Sample   | Total Temp. |
|------|----------------|-----------|----------|-------------|
|      | Heat           | Increase  | Increase | Increase    |
| [s]  | [Jkg-<br>1K-1] | [K]       | [K]      | [K]         |
| 20   | 3989           | 2.01      | 1.97     | 6.9133      |
| 20   | 3753           | 2.01      | 1.98     | 6.9375      |
| 40   | 2166           | 2.39      | 2.39     | 7.1579      |
| 40   | 2096           | 2.39      | 2.39     | 7.1702      |
| 80   | 1523           | 2.20      | 2.22     | 5.9156      |
| 80   | 1669           | 2.20      | 2.65     | 6.9215      |
| 80   | 1178           | 2.62      | 2.66     | 6.9473      |
| 160  | 869            | 2.69      | 2.70     | 6.6880      |
| 160  | 905            | 2.69      | 2.70     | 6.6629      |
| 160  | 1096           | 2.69      | 2.70     | 6.6378      |
| 160  | 855            | 2.69      | 2.70     | 6.6339      |
| 160  | 1174           | 2.69      | 2.69     | 6.6146      |
| 160  | 1107           | 2.69      | 2.69     | 6.6252      |
| 160  | 1023           | 2.69      | 2.69     | 6.6169      |
| 160  | 1130           | 2.69      | 2.69     | 6.6430      |
| 160  | 874            | 2.69      | 2.70     | 6.6550      |
| 160  | 960            | 2.69      | 2.70     | 6.6665      |
| 320  | 2965           | 2.59      | 2.58     | 6.5123      |
| 320  | 2221           | 2.59      | 2.60     | 6.5217      |
| 320  | 1753           | 2.59      | 2.61     | 6.5375      |
| 320  | 2846           | 2.59      | 2.59     | 6.5432      |
| 320  | 685            | 2.59      | 2.64     | 6.5802      |
| 640  | 6886           | 2.30      | 2.26     | 6.5149      |

| 640             | 6766  | 2.30     | 2.26     | 6.5229   |
|-----------------|-------|----------|----------|----------|
| 640             | 3453  | 2.30     | 2.31     | 6.5759   |
| 640             | 4185  | 2.30     | 2.30     | 6.5948   |
| 640             | 310   | 2.30     | 2.34     | 6.6432   |
| 1280            | 24005 | 1.62     | 1.56     | 6.3436   |
| 1280            | 20387 | 1.62     | 1.57     | 6.3903   |
| 1280            | 16392 | 1.62     | 1.59     | 6.4272   |
| 20 [Average]    | 3871  | 2.01     | 1.98     | 6.93     |
| 20 [Std. Dev]   | 166   | 0.00E+00 | 5.76E-03 | 1.71E-02 |
| 40 [Average]    | 2131  | 2.39     | 2.39     | 7.16     |
| 40 [Std. Dev]   | 49    | 0.00E+00 | 4.22E-04 | 8.76E-03 |
| 80 [Average]    | 1457  | 2.34     | 2.51     | 6.59     |
| 80 [Std. Dev]   | 252   | 2.45E-01 | 2.52E-01 | 5.88E-01 |
| 160 [Average]   | 999   | 2.69     | 2.70     | 6.64     |
| 160 [Std. Dev]  | 122   | 4.68E-16 | 4.22E-03 | 2.36E-02 |
| 320 [Average]   | 2094  | 2.59     | 2.60     | 6.54     |
| 320 [Std. Dev]  | 927   | 0.00E+00 | 2.01E-02 | 2.61E-02 |
| 640 [Average]   | 4320  | 2.30     | 2.30     | 6.57     |
| 640 [Std. Dev]  | 2712  | 0.00E+00 | 3.41E-02 | 5.30E-02 |
| 1280 [Average]  | 20261 | 1.62     | 1.57     | 6.39     |
| 1280 [Std. Dev] | 3808  | 0.00E+00 | 1.46E-02 | 4.19E-02 |

|                 |               | RTV-655            |                 |                      |
|-----------------|---------------|--------------------|-----------------|----------------------|
| Time            | Specific Heat | Reference Increase | Sample Increase | Total Temp. Increase |
| [s]             | [Jkg-1K-1]    | [K]                | [K]             | [K]                  |
| 160             | 1274          | 2.64               | 2.79            | 4.07                 |
| 160             | 1268          | 2.64               | 2.97            | 4.33                 |
| 160             | 1295          | 2.64               | 2.95            | 4.30                 |
| 160             | 1285          | 2.64               | 2.96            | 4.30                 |
| 320             | 1443          | 2.79               | 2.84            | 3.92                 |
| 320             | 1465          | 2.79               | 2.82            | 3.88                 |
| 320             | 1456          | 2.79               | 2.94            | 4.05                 |
| 640             | 1407          | 3.07               | 3.17            | 4.23                 |
| 640             | 1321          | 3.07               | 3.23            | 4.30                 |
| 640             | 1379          | 3.07               | 3.19            | 4.28                 |
| 1280            | 610           | 2.92               | 3.49            | 4.75                 |
| 1280            | 585           | 2.92               | 3.51            | 4.74                 |
| 1280            | 526           | 2.92               | 3.54            | 4.78                 |
| 160 [Average]   | 1280          | 2.64               | 2.92            | 4.25                 |
| 160 [Std. Dev]  | 12            | 0.00E+00           | 8.64E-02        | 1.22E-01             |
| 320 [Average]   | 1455          | 2.79               | 2.86            | 3.95                 |
| 320 [Std. Dev]  | 11            | 5.44E-16           | 6.41E-02        | 9.29E-02             |
| 640 [Average]   | 1369          | 3.07               | 3.20            | 4.27                 |
| 640 [Std. Dev]  | 44            | 0.00E+00           | 3.15E-02        | 3.81E-02             |
| 1280 [Average]  | 574           | 2.92               | 3.51            | 4.75                 |
| 1280 [Std. Dev] | 44            | 5.44E-16           | 2.46E-02        | 2.28E-02             |

|--|

| Time | Specific       | Reference | Sample   | Total Temp. |
|------|----------------|-----------|----------|-------------|
|      | Heat           | Increase  | Increase | Increase    |
| [s]  | [Jkg-<br>1K-1] | [K]       | [K]      | [K]         |
| 160  | 1223           | 2.70      | 2.45     | 7.02        |
| 160  | 1229           | 2.70      | 3.11     | 8.91        |
| 160  | 982            | 2.21      | 2.58     | 4.76        |
| 160  | 986            | 2.21      | 2.42     | 4.45        |
| 160  | 991            | 2.21      | 2.42     | 4.43        |
| 160  | 991            | 2.21      | 2.42     | 4.43        |
| 320  | 1601           | 2.59      | 2.33     | 6.53        |
| 320  | 1638           | 2.59      | 2.58     | 7.21        |
| 320  | 1633           | 2.59      | 2.58     | 7.23        |
| 320  | 1627           | 2.59      | 2.58     | 7.25        |
| 320  | 1629           | 2.59      | 2.58     | 7.24        |
| 320  | 1611           | 2.59      | 2.59     | 7.26        |
| 320  | 1593           | 2.59      | 2.61     | 7.30        |
| 320  | 1641           | 2.59      | 2.68     | 7.52        |
| 320  | 1358           | 2.29      | 2.27     | 4.06        |
| 320  | 1325           | 2.29      | 2.34     | 4.16        |
| 320  | 1359           | 2.29      | 2.31     | 4.13        |
| 320  | 1361           | 2.29      | 2.31     | 4.10        |
| 640  | 1620           | 2.30      | 2.31     | 6.45        |
| 640  | 1589           | 2.30      | 2.32     | 6.47        |
| 640  | 1589           | 2.30      | 2.32     | 6.48        |
| 640  | 1556           | 2.30      | 2.34     | 6.52        |
| 640  | 1519           | 2.30      | 2.35     | 6.54        |

| 640             | 1560 | 2.30     | 2.34     | 6.49     |
|-----------------|------|----------|----------|----------|
| 640             | 1721 | 2.30     | 2.27     | 6.36     |
| 640             | 1686 | 2.30     | 2.28     | 6.39     |
| 640             | 1668 | 2.30     | 2.29     | 6.40     |
| 640             | 1665 | 2.30     | 2.29     | 6.41     |
| 640             | 1624 | 2.30     | 2.36     | 6.60     |
| 640             | 1621 | 2.30     | 2.37     | 6.61     |
| 640             | 1564 | 2.30     | 2.45     | 6.81     |
| 640             | 1670 | 2.30     | 2.46     | 6.88     |
| 640             | 1440 | 2.40     | 2.39     | 4.20     |
| 640             | 1480 | 2.40     | 2.37     | 4.17     |
| 640             | 1469 | 2.40     | 2.39     | 4.19     |
| 1280            | 361  | 1.62     | 2.15     | 6.61     |
| 1280            | 639  | 2.04     | 2.45     | 4.42     |
| 1280            | 724  | 2.04     | 2.19     | 3.88     |
| 160 [Average]   | 1067 | 2.37     | 2.57     | 5.67     |
| 160 [Std. Dev]  | 123  | 2.51E-01 | 2.75E-01 | 1.88E+00 |
| 320 [Average]   | 1531 | 2.49     | 2.48     | 6.17     |
| 320 [Std. Dev]  | 134  | 1.47E-01 | 1.52E-01 | 1.53E+00 |
| 640 [Average]   | 1591 | 2.32     | 2.35     | 6.12     |
| 640 [Std. Dev]  | 81   | 3.94E-02 | 5.39E-02 | 9.31E-01 |
| 1280 [Average]  | 575  | 1.90     | 2.26     | 4.97     |
| 1280 [Std. Dev] | 190  | 2.44E-01 | 1.65E-01 | 1.45E+00 |

|      |                  | VR34                  |                    |                         |
|------|------------------|-----------------------|--------------------|-------------------------|
| Time | Specific<br>Heat | Reference<br>Increase | Sample<br>Increase | Total Temp.<br>Increase |
| [s]  | [Jkg-<br>1K-1]   | [K]                   | [K]                | [K]                     |
| 160  | 1107             | 2.21                  | 2.15               | 4.03265                 |
| 160  | 1108             | 2.21                  | 2.18               | 4.09860                 |
| 160  | 1222             | 2.70                  | 2.80               | 7.81007                 |
| 160  | 1115             | 2.21                  | 2.21               | 4.15278                 |
| 320  | 1365             | 2.29                  | 2.14               | 3.80310                 |
| 320  | 2103             | 2.59                  | 2.45               | 6.83732                 |
| 320  | 1555             | 2.59                  | 2.76               | 7.49918                 |
| 320  | 1385             | 2.29                  | 2.20               | 3.89162                 |
| 320  | 1369             | 2.29                  | 2.22               | 3.90153                 |
| 640  | 1662             | 2.30                  | 2.27               | 6.31609                 |
| 640  | 1667             | 2.30                  | 2.29               | 6.36536                 |
| 640  | 1637             | 2.30                  | 2.30               | 6.37052                 |
| 640  | 1669             | 2.30                  | 2.28               | 6.37222                 |
| 640  | 1631             | 2.30                  | 2.30               | 6.39209                 |
| 640  | 1637             | 2.30                  | 2.30               | 6.40041                 |
| 640  | 1643             | 2.30                  | 2.30               | 6.40238                 |
| 640  | 1648             | 2.30                  | 2.29               | 6.40893                 |
| 640  | 1589             | 2.30                  | 2.32               | 6.41657                 |
| 640  | 1601             | 2.30                  | 2.31               | 6.42045                 |
| 640  | 1559             | 2.30                  | 2.43               | 6.71512                 |
| 640  | 1530             | 2.30                  | 2.56               | 7.07350                 |

| 640             | 1419 | 2.40     | 2.31     | 3.98295  |
|-----------------|------|----------|----------|----------|
| 640             | 1424 | 2.40     | 2.34     | 4.02416  |
| 640             | 1510 | 2.40     | 2.33     | 4.03260  |
| 640             | 1492 | 2.40     | 2.34     | 4.04394  |
| 1280            | 381  | 1.62     | 2.05     | 6.43862  |
| 1280            | 880  | 2.04     | 2.08     | 3.69231  |
| 1280            | 793  | 2.04     | 2.05     | 3.60534  |
| 1280            | 1056 | 2.04     | 2.01     | 3.59213  |
| 160 [Average]   | 1138 | 2.33     | 2.34     | 5.02     |
| 160 [Std. Dev]  | 56   | 2.45E-01 | 3.10E-01 | 1.86E+00 |
| 320 [Average]   | 1593 | 2.37     | 2.33     | 5.62     |
| 320 [Std. Dev]  | 245  | 1.36E-01 | 1.96E-01 | 1.51E+00 |
| 640 [Average]   | 1566 | 2.33     | 2.34     | 5.75     |
| 640 [Std. Dev]  | 84   | 4.70E-02 | 7.44E-02 | 1.21E+00 |
| 1280 [Average]  | 778  | 1.93     | 2.05     | 4.33     |
| 1280 [Std. Dev] | 286  | 2.11E-01 | 2.94E-02 | 1.41E+00 |

| VR51 |                |           |          |             |  |  |  |  |  |
|------|----------------|-----------|----------|-------------|--|--|--|--|--|
| Time | Specific       | Reference | Sample   | Total Temp. |  |  |  |  |  |
| Time | Heat           | Increase  | Increase | Increase    |  |  |  |  |  |
| [s]  | [Jkg-<br>1K-1] | [K]       | [K]      | [K]         |  |  |  |  |  |
| 160  | 1150           | 2.70      | 2.83     | 7.78        |  |  |  |  |  |
| 160  | 160 1214 1.4   |           | 1.36     | 4.11        |  |  |  |  |  |
| 320  | 320 1486 1.40  |           | 1.34     | 3.85        |  |  |  |  |  |
| 320  | 1514           | 2.59      | 2.61     | 7.08        |  |  |  |  |  |

| 320 | 1484 | 2.59 | 2.63 | 7.13 |
|-----|------|------|------|------|
| 320 | 1520 | 2.59 | 2.60 | 7.07 |
| 320 | 1593 | 2.59 | 2.56 | 7.00 |
| 320 | 1557 | 2.59 | 2.58 | 7.04 |
| 320 | 1551 | 2.59 | 2.59 | 7.04 |
| 320 | 1538 | 2.59 | 2.59 | 7.06 |
| 320 | 1536 | 2.59 | 2.59 | 7.06 |
| 320 | 1540 | 2.59 | 2.59 | 7.07 |
| 320 | 1520 | 2.59 | 2.60 | 7.07 |
| 320 | 1525 | 2.59 | 2.60 | 7.09 |
| 320 | 1497 | 2.59 | 2.77 | 7.53 |
| 320 | 1549 | 2.59 | 2.78 | 7.56 |
| 640 | 1500 | 2.30 | 2.14 | 5.96 |
| 640 | 1756 | 2.30 | 2.16 | 6.10 |
| 640 | 1482 | 2.30 | 2.33 | 6.50 |
| 640 | 1610 | 2.30 | 2.28 | 6.42 |
| 640 | 1550 | 2.30 | 2.30 | 6.45 |
| 640 | 1538 | 2.30 | 2.31 | 6.47 |
| 640 | 1543 | 2.30 | 2.30 | 6.46 |
| 640 | 1509 | 2.30 | 2.32 | 6.52 |
| 640 | 1687 | 2.30 | 2.31 | 6.48 |
| 640 | 1615 | 2.30 | 2.33 | 6.54 |
| 640 | 1641 | 2.30 | 2.32 | 6.50 |
| 640 | 1639 | 2.30 | 2.32 | 6.50 |

| 640             | 1625 | 2.30     | 2.33     | 6.53     |
|-----------------|------|----------|----------|----------|
| 640             | 1563 | 2.30     | 2.36     | 6.57     |
| 640             | 1580 | 2.30     | 2.35     | 6.56     |
| 640             | 1549 | 2.30     | 2.36     | 6.59     |
| 640             | 1561 | 2.30     | 2.35     | 6.58     |
| 640             | 1589 | 2.30     | 2.34     | 6.58     |
| 640             | 1306 | 1.34     | 1.46     | 4.08     |
| 640             | 1556 | 2.30     | 2.36     | 6.59     |
| 1280            | -471 | 0.93     | 1.25     | 3.71     |
| 1280            | 291  | 1.62     | 2.07     | 6.60     |
| 160 [Average]   | 1182 | 2.05     | 2.10     | 5.94     |
| 160 [Std. Dev]  | 45   | 9.09E-01 | 1.04E+00 | 2.59E+00 |
| 320 [Average]   | 1529 | 2.50     | 2.53     | 6.90     |
| 320 [Std. Dev]  | 30   | 3.16E-01 | 3.49E-01 | 8.95E-01 |
| 640 [Average]   | 1570 | 2.25     | 2.27     | 6.35     |
| 640 [Std. Dev]  | 90   | 2.14E-01 | 1.99E-01 | 5.57E-01 |
| 1280 [Average]  | -90  | 1.27     | 1.66     | 5.15     |
| 1280 [Std. Dev] | 539  | 4.88E-01 | 5.82E-01 | 2.04E+00 |

| PI-Sheets |          |           |          |             |  |  |  |  |  |
|-----------|----------|-----------|----------|-------------|--|--|--|--|--|
| Time      | Specific | Reference | Sample   | Total Temp. |  |  |  |  |  |
|           | Heat     | Increase  | Increase | Increase    |  |  |  |  |  |
| [s]       | [Jkg-    | [K]       | [K]      | [K]         |  |  |  |  |  |
| [0]       | 1K-1]    | [**]      | [**]     | [**]        |  |  |  |  |  |
| 160       | 1304     | 1.69      | 1.71     | 4.45        |  |  |  |  |  |
| 160       | 1304     | 1.69      | 1.71     | 4.45        |  |  |  |  |  |

| 160 | 1213 | 1.69 | 1.73 | 4.45 |
|-----|------|------|------|------|
| 160 | 1216 | 2.63 | 2.67 | 4.45 |
| 160 | 1136 | 2.63 | 2.69 | 4.45 |
| 160 | 1304 | 1.69 | 1.71 | 4.45 |
| 160 | 1213 | 1.69 | 1.73 | 4.45 |
| 320 | 1378 | 2.70 | 1.74 | 2.80 |
| 320 | 1568 | 2.70 | 1.71 | 2.76 |
| 320 | 1578 | 2.70 | 1.70 | 2.74 |
| 320 | 1572 | 2.70 | 1.70 | 2.76 |
| 320 | 1565 | 2.70 | 1.71 | 2.76 |
| 320 | 1378 | 2.70 | 1.74 | 2.80 |
| 320 | 1568 | 2.70 | 1.71 | 2.76 |
| 320 | 1578 | 2.70 | 1.70 | 2.74 |
| 320 | 1572 | 2.70 | 1.70 | 2.76 |
| 320 | 1565 | 2.70 | 1.71 | 2.76 |
| 320 | 1274 | 2.70 | 2.70 | 4.31 |
| 320 | 1274 | 2.70 | 2.70 | 4.31 |
| 320 | 1258 | 2.70 | 2.71 | 4.33 |
| 320 | 1258 | 2.70 | 2.71 | 4.33 |
| 320 | 1448 | 1.67 | 1.67 | 4.29 |
| 320 | 1448 | 1.67 | 1.67 | 4.29 |
| 320 | 1455 | 1.67 | 1.67 | 4.29 |
| 320 | 1397 | 2.70 | 2.67 | 4.29 |
| 320 | 1395 | 2.70 | 2.67 | 4.29 |
| 320 | 1358 | 2.70 | 2.68 | 4.30 |
| 320 | 1350 | 2.70 | 2.68 | 4.29 |

| 320 | 1339 | 2.70 | 2.68 | 4.29 |
|-----|------|------|------|------|
| 320 | 1323 | 2.70 | 2.69 | 4.30 |
| 320 | 1302 | 2.70 | 2.70 | 4.29 |
| 320 | 1448 | 1.67 | 1.67 | 4.29 |
| 320 | 1455 | 1.67 | 1.67 | 4.29 |
| 320 | 1358 | 2.70 | 2.68 | 4.30 |
| 320 | 1350 | 2.70 | 2.68 | 4.29 |
| 320 | 1339 | 2.70 | 2.68 | 4.29 |
| 320 | 1323 | 2.70 | 2.69 | 4.30 |
| 320 | 1302 | 2.70 | 2.70 | 4.29 |
| 320 | 1255 | 2.70 | 2.71 | 4.33 |
| 320 | 1255 | 2.70 | 2.71 | 4.33 |
| 320 | 1352 | 2.70 | 2.68 | 4.29 |
| 320 | 1341 | 2.70 | 2.69 | 4.31 |
| 320 | 1286 | 2.70 | 2.70 | 4.33 |
| 640 | 1911 | 2.40 | 2.28 | 4.30 |
| 640 | 1911 | 2.40 | 2.28 | 4.30 |
| 640 | 1966 | 2.73 | 2.60 | 4.30 |
| 640 | 1675 | 2.73 | 2.66 | 4.39 |
| 640 | 1848 | 2.73 | 2.62 | 4.33 |
| 640 | 1737 | 2.73 | 2.64 | 4.34 |
| 640 | 1455 | 2.73 | 2.71 | 4.43 |
| 640 | 1750 | 2.73 | 2.64 | 4.34 |
| 640 | 1468 | 2.73 | 2.71 | 4.42 |
| 640 | 1524 | 2.73 | 2.69 | 4.41 |
| 640 | 1930 | 2.73 | 2.60 | 4.28 |

| 640 | 2063 | 2.73 | 2.58 | 4.29 |
|-----|------|------|------|------|
| 640 | 1990 | 2.73 | 2.59 | 4.28 |
| 640 | 1911 | 2.40 | 2.28 | 4.30 |
| 640 | 1771 | 1.19 | 1.19 | 4.39 |
| 640 | 1848 | 2.73 | 2.62 | 4.33 |
| 640 | 1737 | 2.73 | 2.64 | 4.34 |
| 640 | 1455 | 2.73 | 2.71 | 4.43 |
| 640 | 1750 | 2.73 | 2.64 | 4.34 |
| 640 | 1468 | 2.73 | 2.71 | 4.42 |
| 640 | 1524 | 2.73 | 2.69 | 4.41 |
| 640 | 1855 | 3.43 | 3.25 | 4.28 |
| 640 | 1965 | 3.43 | 3.22 | 4.29 |
| 640 | 1909 | 3.43 | 3.23 | 4.28 |
| 640 | 1458 | 2.73 | 2.71 | 4.42 |
| 640 | 1458 | 2.73 | 2.71 | 4.42 |
| 640 | 1445 | 2.73 | 2.71 | 4.45 |
| 640 | 1445 | 2.73 | 2.71 | 4.45 |
| 640 | 1367 | 2.73 | 2.74 | 4.49 |
| 640 | 1367 | 2.73 | 2.74 | 4.49 |
| 640 | 1962 | 2.73 | 2.60 | 4.31 |
| 640 | 1880 | 2.73 | 2.62 | 4.34 |
| 640 | 1843 | 2.73 | 2.63 | 4.33 |
| 640 | 1831 | 2.73 | 2.64 | 4.36 |
| 640 | 1850 | 2.73 | 2.63 | 4.35 |
| 640 | 2089 | 1.58 | 1.53 | 4.31 |
| 640 | 1983 | 1.58 | 1.54 | 4.34 |

| 640 | 1935 | 1.58 | 1.55 | 4.33 |
|-----|------|------|------|------|
| 640 | 1921 | 1.58 | 1.55 | 4.36 |
| 640 | 1940 | 1.58 | 1.55 | 4.35 |
| 640 | 1423 | 2.73 | 2.72 | 4.46 |
| 640 | 1423 | 2.73 | 2.72 | 4.46 |
| 640 | 1500 | 3.43 | 3.37 | 4.46 |
| 640 | 1830 | 2.04 | 1.94 | 4.33 |
| 640 | 1534 | 2.73 | 2.70 | 4.46 |
| 640 | 1937 | 2.73 | 2.61 | 4.33 |
| 640 | 1500 | 3.43 | 3.37 | 4.46 |
| 640 | 1830 | 2.04 | 1.94 | 4.33 |
| 640 | 1426 | 3.03 | 3.03 | 4.37 |
| 640 | 1398 | 3.03 | 3.03 | 4.38 |
| 640 | 1368 | 3.31 | 3.31 | 4.36 |
| 640 | 1315 | 3.43 | 3.44 | 4.53 |
| 640 | 1277 | 2.73 | 2.77 | 4.53 |
| 640 | 1625 | 2.73 | 2.70 | 4.43 |
| 640 | 1315 | 3.43 | 3.44 | 4.53 |
| 640 | 1625 | 2.73 | 2.70 | 4.43 |
| 640 | 1824 | 2.73 | 2.65 | 4.35 |
| 640 | 1824 | 2.73 | 2.65 | 4.35 |
| 640 | 1877 | 2.73 | 2.65 | 4.36 |
| 640 | 1877 | 2.73 | 2.65 | 4.36 |
| 640 | 1804 | 2.73 | 2.66 | 4.38 |
| 640 | 1804 | 2.73 | 2.66 | 4.38 |
| 640 | 1780 | 2.73 | 2.67 | 4.42 |

| 640             | 1780 | 2.73     | 2.67     | 4.42     |
|-----------------|------|----------|----------|----------|
| 640             | 1751 | 2.73     | 2.68     | 4.41     |
| 640             | 1792 | 2.73     | 2.68     | 4.34     |
| 640             | 1792 | 2.73     | 2.68     | 4.34     |
| 640             | 1630 | 2.73     | 2.73     | 4.48     |
| 640             | 1608 | 2.73     | 2.73     | 4.47     |
| 640             | 1729 | 2.73     | 2.71     | 4.45     |
| 1280            | 1401 | 1.13     | 1.17     | 4.36     |
| 1280            | 1401 | 1.13     | 1.17     | 4.36     |
| 1280            | 1288 | 1.13     | 1.18     | 4.38     |
| 1280            | 1846 | 2.31     | 2.30     | 4.36     |
| 1280            | 1757 | 2.31     | 2.31     | 4.38     |
| 1280            | 1401 | 1.13     | 1.17     | 4.36     |
| 1280            | 1288 | 1.13     | 1.18     | 4.38     |
| 160 [Average]   | 1242 | 1.96     | 1.99     | 4.45     |
| 160 [Std. Dev]  | 65   | 4.59E-01 | 4.70E-01 | 4.64E-03 |
| 320 [Average]   | 1396 | 2.56     | 2.28     | 3.87     |
| 320 [Std. Dev]  | 110  | 3.59E-01 | 4.96E-01 | 6.98E-01 |
| 640 [Average]   | 1706 | 2.68     | 2.62     | 4.38     |
| 640 [Std. Dev]  | 217  | 4.51E-01 | 4.47E-01 | 6.67E-02 |
| 1280 [Average]  | 1483 | 1.46     | 1.50     | 4.37     |
| 1280 [Std. Dev] | 225  | 5.76E-01 | 5.52E-01 | 1.06E-02 |

## APPENDIX A

## MEASUREMENT RESULTS AT 253K

|        | SS 316      |                      |          |              |             |               |                |                |          |            |           |  |
|--------|-------------|----------------------|----------|--------------|-------------|---------------|----------------|----------------|----------|------------|-----------|--|
| Output | Measurement | Sensor Radius Points | Thermal  | Thermal      | Volumetric  | Drohing Donth | Tamp Ingrades  | Total Temp     | Total to | Mean       |           |  |
| power  | Time        | Selisor Radius       | POINS    | Conductivity | Diffusivity | Specific Heat | Probling Deput | Temp. increase | Increase | Char. Time | Deviation |  |
| [mW]   | [s]         | [mm]                 |          | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]           | [K]            | [K]      |            | [K]       |  |
| 1000   | 20          | 6.403                | 20-100   | 12.940       | 3.561       | 3.634         | 11.9           | 0.411          | 3.08     | 0.867      | 3.28E-05  |  |
| 1000   | 20          | 6.403                | 20-100   | 12.925       | 3.593       | 3.597         | 12.0           | 0.411          | 3.15     | 0.875      | 2.91E-05  |  |
| 1000   | 20          | 6.403                | 20-100   | 12.915       | 3.589       | 3.598         | 12.0           | 0.412          | 3.14     | 0.874      | 2.60E-05  |  |
| 1000   | 20          | 6.403                | 20-100   | 12.954       | 3.493       | 3.709         | 11.8           | 0.411          | 3.05     | 0.851      | 2.63E-04  |  |
| 1000   | 20          | 6.403                | 20-100   | 12.910       | 3.589       | 3.597         | 12.0           | 0.412          | 3.09     | 0.874      | 2.99E-05  |  |
|        |             |                      | Mean     | 12.929       | 3.565       | 3.627         | 11.9           | 0.412          | 3.10     | 0.868      | 7.62E-05  |  |
|        |             |                      | Std. Dev | 1.81E-02     | 4.24E-02    | 4.84E-02      | 7.13E-02       | 3.80E-04       | 4.09E-02 | 1.03E-02   | 1.05E-04  |  |

|        |             |                |          |              | Pol         | ystyrene      |                |                |            |            |           |
|--------|-------------|----------------|----------|--------------|-------------|---------------|----------------|----------------|------------|------------|-----------|
| Output | Measurement | Songor Dadiug  | Dointe   | Thermal      | Thermal     | Volumetric    | Probing Donth  | Tomp Ingrassa  | Total Temp | Total to   | Mean      |
| power  | Time        | Selisor Radius | Folins   | Conductivity | Diffusivity | Specific Heat | Flooling Depui | Temp. Increase | Increase   | Char. Time | Deviation |
| [mW]   | [s]         | [mm]           |          | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]           | [K]            | [K]        |            | [K]       |
| 8      | 40          | 6.403          | 50-115   | 0.024        | 0.593       | 0.040         | 7.4            | 0.811          | 3.22       | 0.332      | 2.92E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.024        | 0.631       | 0.038         | 7.6            | 0.996          | 3.95       | 0.354      | 3.15E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.025        | 0.682       | 0.036         | 7.9            | 0.976          | 3.86       | 0.382      | 3.37E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.024        | 0.625       | 0.038         | 7.6            | 1.001          | 3.97       | 0.350      | 2.79E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.024        | 0.620       | 0.039         | 7.6            | 1.003          | 3.96       | 0.348      | 3.28E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.025        | 0.677       | 0.037         | 7.9            | 0.977          | 3.88       | 0.379      | 3.27E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.025        | 0.662       | 0.037         | 7.8            | 0.982          | 3.92       | 0.371      | 3.31E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.024        | 0.602       | 0.040         | 7.4            | 1.007          | 4.00       | 0.337      | 2.82E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.024        | 0.628       | 0.038         | 7.6            | 0.999          | 3.95       | 0.352      | 3.08E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.025        | 0.681       | 0.036         | 7.9            | 0.976          | 3.89       | 0.382      | 3.65E-04  |
| 10     | 40          | 6.403          | 50-115   | 0.024        | 0.626       | 0.038         | 7.6            | 1.002          | 3.95       | 0.351      | 2.98E-04  |
|        |             |                | Mean     | 0.024        | 0.639       | 0.038         | 7.7            | 0.975          | 3.87       | 0.358      | 3.15E-04  |
|        |             |                | Std. Dev | 4.13E-04     | 3.16E-02    | 1.23E-03      | 1.89E-01       | 5.58E-02       | 2.18E-01   | 1.77E-02   | 2.60E-05  |

|        | RTV-655     |               |          |              |             |               |                |                |            |            |           |
|--------|-------------|---------------|----------|--------------|-------------|---------------|----------------|----------------|------------|------------|-----------|
| Output | Measurement | Sonor Dadius  | Dointe   | Thermal      | Thermal     | Volumetric    | Probing Donth  | Tomp Ingrassa  | Total Temp | Total to   | Mean      |
| power  | Time        | Sensor Radius | Folias   | Conductivity | Diffusivity | Specific Heat | Flooling Depui | Temp. increase | Increase   | Char. Time | Deviation |
| [mW]   | [s]         | [mm]          |          | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]           | [K]            | [K]        |            | [K]       |
| 40     | 320         | 6.403         | 50-150   | 0.174        | 0.156       | 1.115         | 12.2           | 0.815          | 3.29       | 0.911      | 8.06E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.164        | 0.104       | 1.572         | 10.0           | 1.118          | 4.28       | 0.610      | 1.65E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.166        | 0.124       | 1.342         | 10.9           | 1.093          | 4.25       | 0.724      | 4.89E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.171        | 0.146       | 1.172         | 11.8           | 1.047          | 4.19       | 0.852      | 5.51E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.175        | 0.136       | 1.286         | 11.4           | 1.033          | 4.12       | 0.793      | 6.07E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.173        | 0.114       | 1.524         | 10.4           | 1.059          | 4.19       | 0.665      | 2.21E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.168        | 0.095       | 1.773         | 9.5            | 1.100          | 4.26       | 0.553      | 4.87E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.162        | 0.086       | 1.880         | 9.1            | 1.139          | 4.33       | 0.503      | 4.56E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.160        | 0.092       | 1.750         | 9.4            | 1.149          | 4.36       | 0.536      | 1.94E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.163        | 0.108       | 1.510         | 10.2           | 1.124          | 4.32       | 0.632      | 2.06E-04  |
| 40     | 320         | 6.403         | 50-150   | 0.166        | 0.126       | 1.318         | 11.0           | 1.091          | 4.24       | 0.737      | 4.72E-04  |
|        |             |               | Mean     | 0.167        | 0.117       | 1.477         | 10.5           | 1.070          | 4.17       | 0.683      | 4.23E-04  |
|        |             |               | Std. Dev | 5.01E-03     | 2.27E-02    | 2.53E-01      | 1.02E+00       | 9.24E-02       | 2.99E-01   | 1.33E-01   | 2.04E-04  |

|        | VR17        |               |          |              |             |               |               |                |            |            |           |
|--------|-------------|---------------|----------|--------------|-------------|---------------|---------------|----------------|------------|------------|-----------|
| Output | Measurement | Sansor Dadius | Dointa   | Thermal      | Thermal     | Volumetric    | Drohing Donth | Tomp Inoraca   | Total Temp | Total to   | Mean      |
| power  | Time        | Sensor Radius | Pollits  | Conductivity | Diffusivity | Specific Heat | Probing Deput | Temp. increase | Increase   | Char. Time | Deviation |
| [mW]   | [s]         | [mm]          |          | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]          | [K]            | [K]        |            | [K]       |
| 30     | 640         | 6.403         | (25-100) | 0.109        | 0.036       | 3.075         | 6.7           | 1.174          | 4.86       | 0.277      | 1.97E-03  |
| 30     | 640         | 6.403         | (25-120) | 0.116        | 0.025       | 4.702         | 6.2           | 1.191          | 4.87       | 0.231      | 1.66E-03  |
| 30.000 | 640.000     | 6.403         | (25-200) | 0.111        | 0.042       | 2.662         | 10.3          | 1.757          | 5.00       | 0.651      | 8.40E-03  |
| 30     | 640         | 6.403         | (25-200) | 0.106        | 0.030       | 3.596         | 8.7           | 1.792          | 5.03       | 0.461      | 5.98E-03  |
| 30     | 640         | 6.403         | (25-200) | 0.100        | 0.018       | 5.458         | 6.9           | 1.774          | 4.99       | 0.286      | 2.23E-03  |
| 30.000 | 640.000     | 6.403         | (25-200) | 0.113        | 0.051       | 2.212         | 11.4          | 1.738          | 5.02       | 0.793      | 1.08E-02  |
| 30     | 640         | 6.403         | (25-200) | 0.108        | 0.034       | 3.156         | 9.4           | 1.782          | 5.03       | 0.534      | 7.47E-03  |
| 30     | 640         | 6.403         | (25-200) | 0.103        | 0.023       | 4.431         | 7.7           | 1.788          | 5.01       | 0.363      | 4.59E-03  |
| 30.000 | 640.000     | 6.403         | (20-120) | 0.108        | 0.022       | 4.847         | 5.9           | 1.384          | 4.96       | 0.209      | 1.70E-03  |
|        |             |               | Mean     | 0.108        | 0.031       | 3.793         | 8.1           | 1.598          | 4.97       | 0.423      | 4.98E-03  |
|        |             |               | Std. Dev | 4.83E-03     | 1.05E-02    | 1.11E+00      | 1.94E+00      | 2.68E-01       | 6.50E-02   | 2.03E-01   | 3.38E-03  |

|        | VR34        |                |          |              |             |               |                |                |            |            |           |
|--------|-------------|----------------|----------|--------------|-------------|---------------|----------------|----------------|------------|------------|-----------|
| Output | Measurement | Sensor Padius  | Doints   | Thermal      | Thermal     | Volumetric    | Probing Depth  | Temp Increase  | Total Temp | Total to   | Mean      |
| power  | Time        | Selisor Radius | FOILIS   | Conductivity | Diffusivity | Specific Heat | Flooling Deput | Temp. Increase | Increase   | Char. Time | Deviation |
| [mW]   | [s]         | [mm]           |          | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]           | [K]            | [K]        |            | [K]       |
| 70     | 320         | 9.868          | 125-200  | 0.072        | 0.060       | 1.200         | 8.8            | 0.933          | 4.76       | 0.198      | 1.98E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.076        | 0.074       | 1.025         | 9.8            | 0.911          | 4.78       | 0.245      | 1.24E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.072        | 0.112       | 0.644         | 12.0           | 0.988          | 5.16       | 0.369      | 4.34E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.080        | 0.091       | 0.879         | 10.8           | 0.889          | 4.77       | 0.298      | 1.50E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.065        | 0.077       | 0.851         | 9.9            | 1.065          | 5.33       | 0.252      | 3.47E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.067        | 0.054       | 1.249         | 8.3            | 0.981          | 4.95       | 0.177      | 9.38E-05  |
| 70     | 320         | 9.868          | 125-200  | 0.077        | 0.142       | 0.539         | 13.5           | 0.929          | 5.03       | 0.467      | 4.85E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.080        | 0.094       | 0.848         | 11.0           | 0.886          | 4.79       | 0.310      | 1.74E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.064        | 0.068       | 0.934         | 9.3            | 1.080          | 5.32       | 0.224      | 3.53E-04  |
| 70     | 320         | 9.868          | 125-200  | 0.072        | 0.062       | 1.165         | 8.9            | 0.944          | 4.85       | 0.203      | 2.79E-04  |
|        |             |                | Mean     | 0.073        | 0.083       | 0.933         | 10.2           | 0.960          | 4.97       | 0.274      | 2.64E-04  |
|        |             |                | Std. Dev | 5.75E-03     | 2.72E-02    | 2.33E-01      | 1.61E+00       | 6.78E-02       | 2.24E-01   | 8.95E-02   | 1.36E-04  |

|        | VR51        |               |          |           |          |            |               |                |            |          |          |
|--------|-------------|---------------|----------|-----------|----------|------------|---------------|----------------|------------|----------|----------|
| Output | Measurement | Sensor Radius | Points   | Thermal   | Thermal  | Volumetric | Probing Depth | Temp. Increase | Total Temp | Total to | Mean     |
| [mW]   | [s]         | [mm]          |          | [Wm-1K-1] | [mm2s-1] | [MJm-3K-1] | [mm]          | [K]            | [K]        |          | [K]      |
| 55     | 320         | 9.868         | 50-135   | 0.053     | 0.107    | 0.497      | 9.611         | 2.048          | 5.677      | 0.237    | 1.90E-03 |
| 55     | 320         | 9.868         | 75-150   | 0.047     | 0.108    | 0.430      | 10.197        | 1.707          | 6.291      | 0.267    | 9.12E-04 |
| 55     | 320         | 9.868         | 75-150   | 0.049     | 0.101    | 0.487      | 9.861         | 1.604          | 5.973      | 0.250    | 6.69E-04 |
| 55     | 320         | 9.868         | 50-135   | 0.051     | 0.108    | 0.470      | 9.669         | 2.141          | 5.896      | 0.240    | 2.51E-03 |
| 55     | 320         | 9.868         | 75-150   | 0.044     | 0.081    | 0.542      | 8.834         | 1.732          | 6.324      | 0.200    | 6.80E-04 |
| 55     | 320         | 9.868         | 75-150   | 0.053     | 0.122    | 0.435      | 10.810        | 1.525          | 5.763      | 0.300    | 5.75E-04 |
| 55     | 320         | 9.868         | 75-150   | 0.049     | 0.128    | 0.384      | 11.083        | 1.649          | 6.153      | 0.315    | 9.96E-04 |
| 55     | 320         | 9.868         | 75-150   | 0.048     | 0.100    | 0.481      | 9.804         | 1.639          | 6.077      | 0.247    | 4.82E-04 |
| 55     | 320         | 9.868         | 50-135   | 0.053     | 0.122    | 0.437      | 10.276        | 2.080          | 5.757      | 0.271    | 2.83E-03 |
| 55     | 320         | 9.868         | 75-150   | 0.044     | 0.085    | 0.524      | 9.013         | 1.734          | 6.334      | 0.209    | 7.24E-04 |
| 55     | 320         | 9.868         | 75-150   | 0.053     | 0.121    | 0.438      | 10.780        | 1.524          | 5.748      | 0.298    | 5.87E-04 |
| 55     | 320         | 9.868         | 50-135   | 0.048     | 0.095    | 0.506      | 9.067         | 2.209          | 6.068      | 0.211    | 2.43E-03 |
| 55     | 320         | 9.868         | 75-150   | 0.045     | 0.083    | 0.547      | 8.930         | 1.685          | 6.221      | 0.205    | 4.59E-04 |
| 55     | 320         | 9.868         | 50-135   | 0.053     | 0.107    | 0.500      | 9.593         | 2.042          | 5.675      | 0.236    | 1.87E-03 |
|        |             |               | Mean     | 0.049     | 0.105    | 0.477      | 9.823         | 1.809          | 5.997      | 0.249    | 1.26E-03 |
|        |             |               | Std. Dev | 3.44E-03  | 1.51E-02 | 4.71E-02   | 7.31E-01      | 2.40E-01       | 2.46E-01   | 3.72E-02 | 8.54E-04 |

|          |              |             |               | PI       |          |            |                |                |
|----------|--------------|-------------|---------------|----------|----------|------------|----------------|----------------|
|          | Thermal      | Thermal     | Volumetric    | Probing  | Temp.    | Total Temp | Total to Char. | Maan Deviation |
|          | Conductivity | Diffusivity | Specific Heat | Depth    | Increase | Increase   | Time           | Mean Deviation |
|          | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]     | [K]      | [K]        |                | [K]            |
|          | 0.036        | 0.305       | 0.120         | 2.470    | 0.626    | 3.112      | 0.131          | 1.71E-04       |
|          | 0.036        | 0.292       | 0.124         | 2.418    | 0.637    | 3.076      | 0.126          | 1.11E-04       |
|          | 0.036        | 0.321       | 0.113         | 2.532    | 0.642    | 3.121      | 0.138          | 1.44E-04       |
|          | 0.035        | 0.276       | 0.128         | 2.348    | 0.642    | 3.143      | 0.119          | 1.35E-04       |
|          | 0.036        | 0.332       | 0.110         | 2.578    | 0.640    | 3.128      | 0.143          | 2.38E-04       |
|          | 0.036        | 0.319       | 0.114         | 2.524    | 0.637    | 3.126      | 0.137          | 2.42E-04       |
|          | 0.036        | 0.328       | 0.111         | 2.561    | 0.640    | 3.158      | 0.141          | 2.58E-04       |
|          | 0.036        | 0.306       | 0.116         | 2.475    | 0.641    | 3.150      | 0.132          | 1.49E-04       |
|          | 0.036        | 0.319       | 0.114         | 2.525    | 0.641    | 3.099      | 0.137          | 1.53E-04       |
|          | 0.036        | 0.327       | 0.111         | 2.559    | 0.639    | 3.087      | 0.141          | 1.93E-04       |
|          | 0.036        | 0.309       | 0.116         | 2.486    | 0.640    | 3.053      | 0.133          | 2.66E-04       |
|          | 0.036        | 0.319       | 0.113         | 2.526    | 0.643    | 3.118      | 0.137          | 1.95E-04       |
|          | 0.035        | 0.295       | 0.120         | 2.427    | 0.639    | 3.187      | 0.127          | 2.83E-04       |
|          | 0.036        | 0.325       | 0.111         | 2.551    | 0.640    | 3.091      | 0.140          | 2.35E-04       |
|          | 0.036        | 0.300       | 0.119         | 2.448    | 0.637    | 3.150      | 0.129          | 1.35E-04       |
|          | 0.036        | 0.325       | 0.112         | 2.549    | 0.637    | 3.161      | 0.140          | 1.14E-04       |
|          | 0.035        | 0.292       | 0.121         | 2.417    | 0.641    | 3.146      | 0.126          | 1.05E-04       |
| Mean     | 0.036        | 0.311       | 0.116         | 2.494    | 0.639    | 3.124      | 0.134          | 1.84E-04       |
| Std. Dev | 3.82E-04     | 1.60E-02    | 5.26E-03      | 6.48E-02 | 3.77E-03 | 3.47E-02   | 6.90E-03       | 5.94E-05       |

| RTV-655 |               |                       |                 |                         |  |  |  |  |
|---------|---------------|-----------------------|-----------------|-------------------------|--|--|--|--|
| Time    | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |
| [s]     | [Jkg-1K-1]    | [K]                   | [K]             | [K]                     |  |  |  |  |
| 160     | 1995          | 2.71                  | 1.14            | 3.55                    |  |  |  |  |
| 160     | 1467          | 2.71                  | 2.62            | 7.82                    |  |  |  |  |
| 160     | 1321          | 2.71                  | 2.85            | 8.42                    |  |  |  |  |
| 160     | 1430          | 2.71                  | 2.77            | 8.30                    |  |  |  |  |
| 160     | 1485          | 2.71                  | 2.73            | 8.19                    |  |  |  |  |
| 160     | 1491          | 2.71                  | 2.72            | 8.19                    |  |  |  |  |
| 160     | 1392          | 2.71                  | 2.79            | 8.34                    |  |  |  |  |
| 160     | 1514          | 2.71                  | 2.82            | 8.46                    |  |  |  |  |
| 160     | 1514          | 2.71                  | 3.17            | 9.51                    |  |  |  |  |
| 320     | 1308          | 2.38                  | 2.05            | 5.46                    |  |  |  |  |
| 320     | 1344          | 2.55                  | 2.22            | 6.00                    |  |  |  |  |
| 320     | 1527          | 2.38                  | 2.19            | 6.02                    |  |  |  |  |
| 320     | 1402          | 2.55                  | 2.49            | 6.74                    |  |  |  |  |
| 320     | 1645          | 2.55                  | 2.35            | 6.42                    |  |  |  |  |
| 320     | 1321          | 2.55                  | 2.53            | 6.78                    |  |  |  |  |
| 320     | 1552          | 2.55                  | 2.40            | 6.55                    |  |  |  |  |
| 320     | 1440          | 2.55                  | 2.46            | 6.63                    |  |  |  |  |
| 640     | 1333          | 2.34                  | 2.31            | 6.35                    |  |  |  |  |
| 640     | 1419          | 2.75                  | 2.56            | 6.63                    |  |  |  |  |
| 640     | 1562          | 2.34                  | 2.21            | 6.03                    |  |  |  |  |
| 640     | 1551          | 2.75                  | 2.49            | 6.67                    |  |  |  |  |

| 640            | 1561   | 2.34     | 2.22     | 5.99     |
|----------------|--------|----------|----------|----------|
| 640            | 1173   | 2.75     | 3.08     | 7.94     |
| 640            | 1342   | 2.75     | 3.27     | 8.64     |
| 160 [Average]  | 1512   | 2.62     | 2.50     | 7.21     |
| 160 [Std. Dev] | 191.76 | 1.22E-01 | 4.78E-01 | 1.55E+00 |
| 320 [Average]  | 1442   | 2.48     | 2.39     | 6.51     |
| 320 [Std. Dev] | 122.06 | 1.21E-01 | 7.87E-02 | 1.44E-01 |
| 640 [Average]  | 1420   | 2.61     | 2.42     | 6.44     |
| 640 [Std. Dev] | 79.48  | 2.39E-01 | 1.83E-01 | 3.59E-01 |

| VR17 |               |                       |                 |                         |  |  |  |  |
|------|---------------|-----------------------|-----------------|-------------------------|--|--|--|--|
| Time | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |
| [s]  | [Jkg-1K-1]    | [K]                   | [K]             | [K]                     |  |  |  |  |
| 160  | 1072          | 2.71                  | 2.78            | 8.60                    |  |  |  |  |
| 160  | 1050          | 2.71                  | 2.80            | 8.64                    |  |  |  |  |
| 160  | 1137          | 2.71                  | 2.72            | 8.46                    |  |  |  |  |
| 160  | 1065          | 2.71                  | 2.78            | 8.61                    |  |  |  |  |
| 160  | 1099          | 2.71                  | 2.75            | 8.53                    |  |  |  |  |
| 160  | 1068          | 2.71                  | 2.95            | 9.15                    |  |  |  |  |
| 320  | 1605          | 2.55                  | 2.35            | 7.04                    |  |  |  |  |
| 320  | 1375          | 2.55                  | 2.66            | 7.86                    |  |  |  |  |
| 320  | 1623          | 2.55                  | 2.49            | 7.45                    |  |  |  |  |

| 320            | 1522   | 2.55     | 2.56     | 7.64     |
|----------------|--------|----------|----------|----------|
| 320            | 1438   | 2.55     | 2.61     | 7.70     |
| 320            | 1668   | 2.55     | 2.46     | 7.41     |
| 640            | 1243   | 2.34     | 2.41     | 6.91     |
| 640            | 1338   | 2.34     | 2.36     | 6.63     |
| 640            | 1336   | 2.34     | 2.48     | 7.23     |
| 160 [Average]  | 1082   | 2.71     | 2.80     | 8.67     |
| 160 [Std. Dev] | 31.35  | 0.00E+00 | 8.22E-02 | 2.49E-01 |
| 320 [Average]  | 1538   | 2.55     | 2.52     | 7.52     |
| 320 [Std. Dev] | 114.47 | 0.00E+00 | 1.13E-01 | 2.88E-01 |
| 640 [Average]  | 1306   | 2.34     | 2.42     | 6.92     |
| 640 [Std. Dev] | 53.93  | 0.00E+00 | 6.35E-02 | 3.04E-01 |

| VR34 |                  |                       |                 |                         |  |  |  |  |
|------|------------------|-----------------------|-----------------|-------------------------|--|--|--|--|
| Time | Specific<br>Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |
| [8]  | [Jkg-1K-1]       | [K]                   | [K]             | [K]                     |  |  |  |  |
| 160  | 1161             | 2.71                  | 2.75            | 8.78                    |  |  |  |  |
| 160  | 1200             | 2.71                  | 2.71            | 8.70                    |  |  |  |  |
| 160  | 1201             | 2.71                  | 2.71            | 8.71                    |  |  |  |  |
| 160  | 1212             | 2.71                  | 2.71            | 8.69                    |  |  |  |  |
| 160  | 1206             | 2.71                  | 2.71            | 8.70                    |  |  |  |  |
| 160  | 1210             | 2.71                  | 2.71            | 8.69                    |  |  |  |  |
| 320  | 1331             | 2.55                  | 2.69            | 7.89                    |  |  |  |  |
| 320  | 1539             | 2.55                  | 2.55            | 7.57                    |  |  |  |  |

| 320            | 1636   | 2.55     | 2.49     | 7.50     |
|----------------|--------|----------|----------|----------|
| 320            | 1488   | 2.55     | 2.58     | 7.69     |
| 320            | 1379   | 2.55     | 2.66     | 7.84     |
| 320            | 1460   | 2.55     | 2.64     | 7.81     |
| 640            | 1481   | 2.34     | 2.25     | 6.65     |
| 640            | 1556   | 2.34     | 2.28     | 6.56     |
| 640            | 1764   | 2.34     | 2.19     | 6.42     |
| 640            | 1843   | 2.34     | 2.16     | 6.43     |
| 640            | 1860   | 2.34     | 2.15     | 6.45     |
| 640            | 1829   | 2.34     | 2.16     | 6.51     |
| 160 [Average]  | 1198   | 2.71     | 2.72     | 8.71     |
| 160 [Std. Dev] | 19.06  | 0.00E+00 | 1.49E-02 | 3.28E-02 |
| 320 [Average]  | 1472   | 2.55     | 2.60     | 7.72     |
| 320 [Std. Dev] | 109.78 | 0.00E+00 | 7.40E-02 | 1.58E-01 |
| 640 [Average]  | 1722   | 2.34     | 2.20     | 6.51     |
| 640 [Std. Dev] | 162.85 | 4.86E-16 | 5.16E-02 | 8.94E-02 |

| VR51 |                  |                       |                 |                         |  |  |  |  |
|------|------------------|-----------------------|-----------------|-------------------------|--|--|--|--|
| Time | Specific<br>Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |
| [s]  | [Jkg-1K-1]       | [K]                   | [K]             | [K]                     |  |  |  |  |
| 160  | 1138             | 2.71                  | 2.74            | 9.96                    |  |  |  |  |
| 160  | 1134             | 2.71                  | 2.74            | 9.97                    |  |  |  |  |
| 160  | 1105             | 2.71                  | 2.76            | 10.02                   |  |  |  |  |
| 160  | 1057             | 2.71                  | 2.80            | 10.12                   |  |  |  |  |

| 160            | 1010   | 2.71     | 2.84     | 10.23    |
|----------------|--------|----------|----------|----------|
| 160            | 1036   | 2.71     | 2.93     | 10.46    |
| 320            | 1296   | 2.55     | 2.69     | 8.73     |
| 320            | 1382   | 2.55     | 2.64     | 8.57     |
| 320            | 1489   | 2.55     | 2.57     | 8.45     |
| 320            | 1571   | 2.55     | 2.52     | 8.33     |
| 320            | 1427   | 2.55     | 2.69     | 8.83     |
| 640            | 1956   | 2.34     | 2.17     | 7.05     |
| 640            | 1149   | 2.34     | 2.71     | 8.26     |
| 640            | 1297   | 2.34     | 2.64     | 8.31     |
| 640            | 1642   | 2.34     | 2.48     | 8.04     |
| 640            | 1968   | 2.34     | 2.34     | 7.69     |
| 640            | 1410   | 2.34     | 2.58     | 7.96     |
| 160 [Average]  | 1080   | 2.37     | 2.52     | 8.02     |
| 160 [Std. Dev] | 53.28  | 7.92E-02 | 2.01E-01 | 5.54E-01 |
| 320 [Average]  | 1433   | 2.529    | 2.64     | 8.88     |
| 320 [Std. Dev] | 104.43 | 1.61E-01 | 1.87E-01 | 1.03E+00 |
| 640 [Average]  | 1570   | 2.354    | 2.48     | 8.21     |
| 640 [Std. Dev] | 343.42 | 6.05E-01 | 6.15E-01 | 2.20E+00 |

| PI-Sheets |                  |                       |                 |                         |  |  |  |  |  |  |  |
|-----------|------------------|-----------------------|-----------------|-------------------------|--|--|--|--|--|--|--|
| Time      | Specific<br>Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |  |  |  |
| [s]       | [Jkg-1K-1]       | [K]                   | [K]             | [K]                     |  |  |  |  |  |  |  |
| 160       | 1491             | 2.67                  | 2.56            | 6.53                    |  |  |  |  |  |  |  |

| 160            | 1709   | 2.71     | 2.65     | 6.73     |
|----------------|--------|----------|----------|----------|
| 160            | 1366   | 2.71     | 2.69     | 6.86     |
| 160            | 910    | 2.71     | 2.76     | 7.01     |
| 160            | 1273   | 2.71     | 2.71     | 6.87     |
| 160            | 1752   | 2.71     | 2.64     | 6.73     |
| 160            | 1325   | 2.67     | 2.67     | 6.78     |
| 160            | 1287   | 2.67     | 2.67     | 6.80     |
| 160            | 916    | 2.71     | 2.76     | 7.02     |
| 160            | 1199   | 2.71     | 2.72     | 6.88     |
| 160            | 1411   | 2.67     | 2.65     | 6.77     |
| 160            | 1106   | 2.71     | 2.74     | 6.97     |
| 320            | 1688   | 2.55     | 2.55     | 6.40     |
| 320            | 2210   | 2.55     | 2.49     | 6.34     |
| 320            | 325    | 2.55     | 2.71     | 6.80     |
| 320            | 2189   | 2.55     | 2.49     | 6.28     |
| 320            | 1226   | 2.55     | 2.60     | 6.62     |
| 320            | 1560   | 2.55     | 2.56     | 6.41     |
| 320            | 1639   | 2.55     | 2.55     | 6.40     |
| 320            | 1545   | 2.55     | 2.57     | 6.42     |
| 320            | 1429   | 2.55     | 2.58     | 6.45     |
| 320            | 1264   | 2.55     | 2.60     | 6.47     |
| 160 [Average]  | 1312   | 2.69     | 2.68     | 6.83     |
| 160 [Std. Dev] | 264.86 | 2.06E-02 | 5.75E-02 | 1.38E-01 |
| 320 [Average]  | 1507   | 2.55     | 2.57     | 6.46     |
| 320 [Std. Dev] | 532.76 | 4.68E-16 | 6.23E-02 | 1.50E-01 |

## APPENDIX A

## MEASUREMENT RESULTS AT 85K

|        |             |        |           |              |             | SS 316        |         |          |            |                |                |
|--------|-------------|--------|-----------|--------------|-------------|---------------|---------|----------|------------|----------------|----------------|
| Output | Measurement | Sensor | Dointa    | Thermal      | Thermal     | Volumetric    | Probing | Temp.    | Total Temp | Total to Char. | Maan Daviation |
| power  | Time        | Radius | Folits    | Conductivity | Diffusivity | Specific Heat | Depth   | Increase | Increase   | Time           | Wean Deviation |
| [mW]   | [s]         | [mm]   |           | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]    | [K]      | [K]        |                | [K]            |
| 200.0  | 20          | 9.868  | 25-105    | 8.326        | 4.927       | 1.690         | 14.4    | 0.074    | 0.51       | 0.531          | 1.69E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.294        | 4.705       | 1.763         | 14.1    | 0.280    | 1.95       | 0.507          | 1.75E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.313        | 4.716       | 1.763         | 14.1    | 0.279    | 1.95       | 0.509          | 1.24E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.293        | 4.716       | 1.759         | 14.1    | 0.280    | 1.95       | 0.509          | 1.97E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.303        | 4.713       | 1.762         | 14.1    | 0.280    | 1.95       | 0.508          | 1.87E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.304        | 4.723       | 1.758         | 14.1    | 0.280    | 1.95       | 0.509          | 1.40E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.309        | 4.717       | 1.762         | 14.1    | 0.280    | 1.95       | 0.509          | 1.67E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.311        | 4.714       | 1.763         | 14.1    | 0.280    | 1.95       | 0.508          | 1.60E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.337        | 4.749       | 1.756         | 14.1    | 0.279    | 1.94       | 0.512          | 1.94E-05       |
| 750.0  | 20          | 9.868  | 25-105    | 8.376        | 4.762       | 1.759         | 14.1    | 0.277    | 1.94       | 0.514          | 1.41E-05       |
|        |             |        | Mean      | 8.317        | 4.744       | 1.753         | 14.1    | 0.259    | 1.80       | 0.512          | 1.65E-05       |
|        |             |        | Std. Dev. | 0.025        | 0.067       | 0.022         | 0.1     | 0.065    | 0.45       | 0.007          | 2.43E-06       |

|        |             |        |           |              | Polys       | styrene (XPS) 8 | 35 Kelvin |          |            |                |                |
|--------|-------------|--------|-----------|--------------|-------------|-----------------|-----------|----------|------------|----------------|----------------|
| Output | Measurement | Sensor | Dointa    | Thermal      | Thermal     | Volumetric      | Probing   | Temp.    | Total Temp | Total to Char. | Maan Daviation |
| power  | Time        | Radius | Points    | Conductivity | Diffusivity | Specific Heat   | Depth     | Increase | Increase   | Time           | Mean Deviation |
| [mW]   | [s]         | [mm]   |           | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]      | [mm]      | [K]      | [K]        |                | [K]            |
| 2.5    | 160         | 6.403  | 15-30     | 0.010        | 1.001       | 0.010           | 9.802     | 0.416    | 2.521      | 0.585          | 0.000          |
| 2.5    | 160         | 6.403  | 15-30     | 0.010        | 1.022       | 0.010           | 9.907     | 0.423    | 2.519      | 0.598          | 0.000          |
| 2.5    | 160         | 6.403  | 15-30     | 0.010        | 1.014       | 0.010           | 9.868     | 0.423    | 2.519      | 0.593          | 0.000          |
| 2.5    | 160         | 6.403  | 15-30     | 0.010        | 1.037       | 0.010           | 9.976     | 0.423    | 2.529      | 0.606          | 0.000          |
| 2.5    | 160         | 6.403  | 15-30     | 0.010        | 1.031       | 0.010           | 9.947     | 0.421    | 2.523      | 0.603          | 0.000          |
| 2.5    | 160         | 6.403  | 15-30     | 0.010        | 0.995       | 0.010           | 9.774     | 0.423    | 2.540      | 0.582          | 0.000          |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.747       | 0.014           | 7.7       | 0.774    | 3.39       | 0.364          | 3.24E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.757       | 0.013           | 7.8       | 0.772    | 3.40       | 0.369          | 2.78E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.763       | 0.013           | 7.8       | 0.774    | 3.38       | 0.372          | 2.89E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.744       | 0.014           | 7.7       | 0.774    | 3.41       | 0.362          | 3.23E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.752       | 0.014           | 7.8       | 0.771    | 3.41       | 0.366          | 3.32E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.750       | 0.014           | 7.7       | 0.770    | 3.41       | 0.365          | 2.69E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.744       | 0.014           | 7.7       | 0.762    | 3.38       | 0.363          | 3.82E-04       |
| 5      | 20          | 6.403  | 100-200   | 0.010        | 0.751       | 0.014           | 7.7       | 0.767    | 3.37       | 0.366          | 4.11E-04       |
|        |             |        | Mean      | 0.010        | 0.751       | 0.013           | 7.750     | 0.771    | 3.394      | 0.366          | 0.000          |
|        |             |        | Std. Dev. | 4.08E-05     | 6.59E-03    | 1.64E-04        | 3.40E-02  | 4.13E-03 | 1.55E-02   | 3.21E-03       | 4.97E-05       |

|        | RTV-655     |        |           |              |             |               |          |          |            |                |                |  |
|--------|-------------|--------|-----------|--------------|-------------|---------------|----------|----------|------------|----------------|----------------|--|
| Output | Measurement | Sensor | Dointa    | Thermal      | Thermal     | Volumetric    | Probing  | Temp.    | Total Temp | Total to Char. | Maan Deviation |  |
| power  | Time        | Radius | Points    | Conductivity | Diffusivity | Specific Heat | Depth    | Increase | Increase   | Time           | Mean Deviation |  |
| [mW]   | [s]         | [mm]   |           | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]     | [K]      | [K]        |                | [K]            |  |
| 50     | 80          | 6.403  | 50-200    | 0.117        | 0.374       | 0.314         | 10.937   | 2.008    | 9.114      | 0.729          | 0.00           |  |
| 50     | 80          | 6.403  | 50-200    | 0.117        | 0.370       | 0.317         | 10.887   | 2.006    | 9.107      | 0.722          | 0.00           |  |
| 50     | 80          | 6.403  | 50-200    | 0.118        | 0.370       | 0.318         | 10.887   | 2.002    | 9.078      | 0.722          | 0.00           |  |
| 50     | 80          | 6.403  | 50-200    | 0.118        | 0.370       | 0.318         | 10.883   | 2.003    | 9.078      | 0.721          | 0.00           |  |
| 50     | 80          | 6.403  | 50-200    | 0.117        | 0.384       | 0.306         | 11.084   | 1.998    | 9.066      | 0.748          | 0.00           |  |
| 40     | 160         | 6.403  | 20-130    | 0.116        | 0.371       | 0.311         | 12.429   | 2.187    | 7.961      | 0.941          | 0.00           |  |
| 40     | 160         | 6.403  | 20-130    | 0.116        | 0.371       | 0.311         | 12.427   | 2.189    | 7.968      | 0.940          | 0.00           |  |
| 40     | 160         | 6.403  | 20-130    | 0.115        | 0.371       | 0.311         | 12.430   | 2.190    | 7.968      | 0.941          | 0.00           |  |
| 40     | 160         | 6.403  | 20-130    | 0.116        | 0.371       | 0.312         | 12.418   | 2.193    | 7.982      | 0.939          | 0.00           |  |
| 40     | 160         | 6.403  | 20-130    | 0.115        | 0.371       | 0.311         | 12.425   | 2.192    | 7.965      | 0.940          | 0.00           |  |
| 30     | 320         | 6.403  | (10-50)   | 0.113        | 0.385       | 0.294         | 11.102   | 1.504    | 6.426      | 0.751          | 0.00           |  |
| 30     | 320         | 6.403  | (10-50)   | 0.113        | 0.386       | 0.293         | 11.108   | 1.503    | 6.426      | 0.752          | 0.00           |  |
| 30     | 320         | 6.403  | (10-50)   | 0.113        | 0.385       | 0.293         | 11.103   | 1.504    | 6.421      | 0.751          | 0.00           |  |
| 30     | 320         | 6.403  | (10-50)   | 0.113        | 0.373       | 0.303         | 10.920   | 1.509    | 6.427      | 0.726          | 0.00           |  |
| 30     | 320         | 6.403  | (10-50)   | 0.113        | 0.385       | 0.293         | 11.102   | 1.504    | 6.418      | 0.751          | 0.00           |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.401       | 0.274         | 7.601    | 0.488    | 3.183      | 0.352          | 8.69E-05       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.403       | 0.272         | 7.615    | 0.489    | 3.188      | 0.353          | 8.36E-05       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.400       | 0.274         | 7.586    | 0.489    | 3.191      | 0.350          | 1.35E-04       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.399       | 0.275         | 7.576    | 0.489    | 3.194      | 0.350          | 9.47E-05       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.398       | 0.276         | 7.572    | 0.488    | 3.189      | 0.349          | 1.20E-04       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.399       | 0.275         | 7.581    | 0.489    | 3.190      | 0.350          | 9.95E-05       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.404       | 0.271         | 7.632    | 0.489    | 3.187      | 0.355          | 1.13E-04       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.402       | 0.273         | 7.609    | 0.488    | 3.179      | 0.353          | 1.06E-04       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.400       | 0.274         | 7.591    | 0.488    | 3.188      | 0.351          | 8.65E-05       |  |
| 20     | 40          | 6.403  | 80-180    | 0.110        | 0.400       | 0.275         | 7.588    | 0.488    | 3.177      | 0.351          | 8.88E-05       |  |
|        |             |        | Mean      | 0.113        | 0.386       | 0.294         | 9.924    | 1.335    | 5.971      | 0.623          | 0.001          |  |
|        |             |        | Std. Dev. | 3.14E-03     | 1.34E-02    | 1.80E-02      | 2.01E+00 | 7.42E-01 | 2.48E+00   | 2.39E-01       | 5.41E-04       |  |

|        | VR17        |        |           |              |             |               |          |          |            |                |                 |  |  |  |
|--------|-------------|--------|-----------|--------------|-------------|---------------|----------|----------|------------|----------------|-----------------|--|--|--|
| Output | Measurement | Sensor | Dointe    | Thermal      | Thermal     | Volumetric    | Probing  | Temp.    | Total Temp | Total to Char. | Mean Deviation  |  |  |  |
| power  | Time        | Radius | Folits    | Conductivity | Diffusivity | Specific Heat | Depth    | Increase | Increase   | Time           | Weall Deviation |  |  |  |
| [mW]   | [s]         | [mm]   |           | [Wm-1K-1]    | [mm2s-1]    | [MJm-3K-1]    | [mm]     | [K]      | [K]        |                | [K]             |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.089        | 0.063       | 1.412         | 8.996    | 0.319    | 3.178      | 0.493          | 0.000           |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.090        | 0.078       | 1.146         | 10.014   | 0.317    | 3.174      | 0.611          | 0.001           |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.090        | 0.065       | 1.373         | 9.136    | 0.318    | 3.175      | 0.508          | 0.000           |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.089        | 0.066       | 1.358         | 9.172    | 0.319    | 3.186      | 0.512          | 0.000           |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.089        | 0.065       | 1.368         | 9.151    | 0.318    | 3.181      | 0.510          | 0.000           |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.090        | 0.066       | 1.348         | 9.220    | 0.318    | 3.182      | 0.518          | 0.000           |  |  |  |
| 12     | 320         | 6.403  | (100-200) | 0.089        | 0.066       | 1.362         | 9.165    | 0.318    | 3.180      | 0.512          | 0.000           |  |  |  |
| 8      | 640         | 6.403  | (80-195)  | 0.091        | 0.057       | 1.582         | 11.964   | 0.257    | 2.335      | 0.872          | 0.000           |  |  |  |
| 8      | 640         | 6.403  | (80-200)  | 0.092        | 0.055       | 1.670         | 11.856   | 0.262    | 2.330      | 0.856          | 0.000           |  |  |  |
| 8      | 640         | 6.403  | (80-191)  | 0.091        | 0.059       | 1.552         | 11.967   | 0.250    | 2.333      | 0.872          | 0.000           |  |  |  |
| 8      | 640         | 6.403  | (80-193)  | 0.091        | 0.058       | 1.561         | 11.986   | 0.253    | 2.332      | 0.875          | 1.03E-04        |  |  |  |
| 8      | 640         | 6.403  | (80-197)  | 0.091        | 0.057       | 1.613         | 11.957   | 0.257    | 2.324      | 0.871          | 1.02E-04        |  |  |  |
| 8      | 640         | 6.403  | (80-199)  | 0.091        | 0.057       | 1.617         | 12.000   | 0.261    | 2.332      | 0.877          | 1.01E-04        |  |  |  |
| 8      | 640         | 6.403  | (80-195)  | 0.091        | 0.058       | 1.575         | 12.021   | 0.255    | 2.321      | 0.880          | 1.06E-04        |  |  |  |
| 8      | 640         | 6.403  | (80-200)  | 0.091        | 0.056       | 1.641         | 11.928   | 0.263    | 2.325      | 0.867          | 1.08E-04        |  |  |  |
| 8      | 640         | 6.403  | (80-198)  | 0.091        | 0.057       | 1.600         | 12.018   | 0.259    | 2.322      | 0.880          | 1.05E-04        |  |  |  |
| 8      | 640         | 6.403  | (80-200)  | 0.092        | 0.056       | 1.642         | 11.950   | 0.262    | 2.324      | 0.870          | 1.02E-04        |  |  |  |
|        |             |        | Mean      | 0.090        | 0.061       | 1.495         | 10.853   | 0.283    | 2.678      | 0.728          | 0.000           |  |  |  |
|        |             |        | Std. Dev. | 9.28E-04     | 6.13E-03    | 1.49E-01      | 1.39E+00 | 3.09E-02 | 4.32E-01   | 1.79E-01       | 2.76E-04        |  |  |  |

|                 | VR34                |                  |        |                         |                        |                                |                  |                   |                           |                           |                   |  |  |
|-----------------|---------------------|------------------|--------|-------------------------|------------------------|--------------------------------|------------------|-------------------|---------------------------|---------------------------|-------------------|--|--|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific<br>Heat | Probing<br>Depth | Temp.<br>Increase | Total<br>Temp<br>Increase | Total to<br>Char.<br>Time | Mean<br>Deviation |  |  |

| [mW]   | [s]     | [mm]  |                     | [Wm-1K-1] | [mm2s-1] | [MJm-3K-<br>1] | [mm]   | [K]   | [K]   |       | [K]   |
|--------|---------|-------|---------------------|-----------|----------|----------------|--------|-------|-------|-------|-------|
| 10.000 | 640.000 | 6.403 | (100-184)           | 0.076     | 0.061    | 1.254          | 11.955 | 0.252 | 2.113 | 0.870 | 0.001 |
| 18.000 | 640.000 | 6.403 | (100-176)           | 0.068     | 0.064    | 1.059          | 12.032 | 0.472 | 4.071 | 0.882 | 0.000 |
| 18.000 | 640.000 | 6.403 | (50-146)            | 0.065     | 0.076    | 0.847          | 11.952 | 0.974 | 4.111 | 0.870 | 0.001 |
| 18.000 | 640.000 | 6.403 | (100-167)           | 0.066     | 0.068    | 0.975          | 12.028 | 0.440 | 4.145 | 0.881 | 0.000 |
| 18.000 | 640.000 | 6.403 | (100-159)           | 0.065     | 0.071    | 0.916          | 12.026 | 0.402 | 4.144 | 0.881 | 0.000 |
| 18.000 | 640.000 | 6.403 | (100-165)           | 0.066     | 0.068    | 0.961          | 12.026 | 0.431 | 4.137 | 0.881 | 0.000 |
| 18.000 | 640.000 | 6.403 | (100-163)           | 0.066     | 0.069    | 0.944          | 12.035 | 0.421 | 4.137 | 0.882 | 0.000 |
| 18.000 | 640.000 | 6.403 | (100-163)           | 0.065     | 0.069    | 0.945          | 12.015 | 0.422 | 4.149 | 0.879 | 0.000 |
| 18.000 | 640.000 | 6.403 | (100-167)           | 0.066     | 0.067    | 0.981          | 11.967 | 0.443 | 4.155 | 0.872 | 0.000 |
| 18.000 | 640.000 | 6.403 | (100-165)           | 0.065     | 0.068    | 0.955          | 12.026 | 0.433 | 4.148 | 0.881 | 0.000 |
| 18     | 640     | 6.403 | (100-182)           | 0.067     | 0.062    | 1.081          | 12.0   | 0.512 | 4.15  | 0.875 | 0.000 |
| 24     | 320     | 6.403 | (100-<br>200,tc,td) | 0.060     | 0.069    | 0.876          | 9.4    | 0.928 | 4.86  | 0.535 | 0.000 |
| 24     | 320     | 6.403 | (100-<br>200,tc,td) | 0.060     | 0.070    | 0.859          | 9.5    | 0.923 | 4.85  | 0.548 | 0.000 |
| 24     | 320     | 6.403 | (100-<br>200,tc,td) | 0.060     | 0.068    | 0.887          | 9.3    | 0.933 | 4.89  | 0.527 | 0.000 |
| 24     | 320     | 6.403 | (100-<br>200,tc,td) | 0.060     | 0.068    | 0.885          | 9.3    | 0.933 | 4.89  | 0.528 | 0.000 |
| 24     | 320     | 6.403 | (100-<br>200,tc,td) | 0.060     | 0.071    | 0.856          | 9.5    | 0.923 | 4.84  | 0.550 | 0.000 |
| 20     | 640     | 6.403 | (57-199)            | 0.064     | 0.056    | 1.145          | 12.0   | 1.282 | 4.64  | 0.871 | 0.001 |
| 20     | 640     | 6.403 | (57-200)            | 0.064     | 0.056    | 1.149          | 12.0   | 1.287 | 4.64  | 0.871 | 0.001 |
| 20     | 640     | 6.403 | (57-200)            | 0.064     | 0.056    | 1.150          | 12.0   | 1.287 | 4.63  | 0.871 | 0.001 |
| 20     | 640     | 6.403 | (57-200)            | 0.064     | 0.056    | 1.146          | 12.0   | 1.288 | 4.64  | 0.873 | 0.001 |

| 20 | 640  | 6.403 | (57-200)           | 0.064    | 0.056    | 1.138    | 12.0     | 1.288        | 4.64     | 0.877        | 0.001    |
|----|------|-------|--------------------|----------|----------|----------|----------|--------------|----------|--------------|----------|
| 16 | 1280 | 6.403 | (50-<br>96,tc,td)  | 0.069    | 0.058    | 1.181    | 12.0     | 0.491        | 4.06     | 0.871        | 0.000    |
| 16 | 1280 | 6.403 | (50-<br>115,tc,td) | 0.069    | 0.049    | 1.404    | 12.0     | 0.631        | 4.13     | 0.879        | 0.000    |
| 16 | 1280 | 6.403 | (50-<br>117,tc,td) | 0.070    | 0.048    | 1.442    | 12.0     | 0.638        | 4.12     | 0.880        | 0.000    |
| 16 | 1280 | 6.403 | (50-<br>163,tc,td) | 0.068    | 0.035    | 1.974    | 12.0     | 0.917        | 4.25     | 0.879        | 0.001    |
| 16 | 1280 | 6.403 | (50-<br>98,tc,td)  | 0.068    | 0.057    | 1.184    | 12.0     | 0.513        | 4.11     | 0.876        | 0.000    |
|    |      |       | Mean               | 0.065    | 0.062    | 1.084    | 11.496   | 0.749        | 4.293    | 0.811        | 0.000    |
|    |      |       | Std. Dev.          | 3.59E-03 | 9.21E-03 | 2.44E-01 | 1.05E+00 | 3.43E-<br>01 | 5.43E-01 | 1.36E-<br>01 | 3.95E-04 |

|                 |                     |                  |        |                         | VR                     | .51                            |                  |                   |                           |                           |                   |
|-----------------|---------------------|------------------|--------|-------------------------|------------------------|--------------------------------|------------------|-------------------|---------------------------|---------------------------|-------------------|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific<br>Heat | Probing<br>Depth | Temp.<br>Increase | Total<br>Temp<br>Increase | Total to<br>Char.<br>Time | Mean<br>Deviation |
| [mW]            | [s]                 | [mm]             |        | [Wm-1K-1]               | [mm2s-1]               | [MJm-3K-<br>1]                 | [mm]             | [K]               | [K]                       |                           | [K]               |
| 10              | 320                 | 6.403            | 75-122 | 0.028                   | 0.184                  | 0.153                          | 11.987           | 0.542             | 3.772                     | 0.875                     | 0.000             |
| 10.3            | 320                 | 6.403            | 75-125 | 0.028                   | 0.181                  | 0.155                          | 12.036           | 0.587             | 3.892                     | 0.882                     | 0.000             |
| 10.3            | 320                 | 6.403            | 75-122 | 0.028                   | 0.183                  | 0.153                          | 11.965           | 0.558             | 3.858                     | 0.872                     | 0.000             |
| 10.3            | 320                 | 6.403            | 75-120 | 0.028                   | 0.188                  | 0.150                          | 12.016           | 0.537             | 3.889                     | 0.879                     | 0.000             |
| 10.3            | 320                 | 6.403            | 75-121 | 0.028                   | 0.187                  | 0.150                          | 12.032           | 0.548             | 3.869                     | 0.882                     | 0.000             |
| 10.2 | 220 | 6 102 | 75 100       | 0.020    | 0.194    | 0.154    | 11 077   | 0.557        | 2 962    | 0.074    | 0.000    |
|------|-----|-------|--------------|----------|----------|----------|----------|--------------|----------|----------|----------|
| 10.5 | 320 | 0.403 | /3-122       | 0.028    | 0.184    | 0.154    | 11.977   | 0.557        | 3.802    | 0.874    | 0.000    |
| 10.3 | 320 | 6.403 | 74-123       | 0.028    | 0.183    | 0.155    | 12.000   | 0.581        | 3.898    | 0.877    | 0.000    |
| 10.3 | 320 | 6.403 | 75-126       | 0.028    | 0.179    | 0.158    | 12.000   | 0.596        | 3.914    | 0.877    | 0.000    |
| 10.3 | 320 | 6.403 | 75-121       | 0.028    | 0.186    | 0.152    | 11.996   | 0.548        | 3.873    | 0.876    | 0.000    |
| 10.3 | 320 | 6.403 | 75-123       | 0.028    | 0.184    | 0.153    | 12.021   | 0.568        | 3.877    | 0.880    | 0.000    |
| 10.3 | 320 | 6.403 | 75-122       | 0.028    | 0.186    | 0.152    | 12.042   | 0.558        | 3.869    | 0.883    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.092    | 0.312    | 6.426    | 0.888        | 3.898    | 0.251    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.094    | 0.309    | 6.476    | 0.885        | 3.871    | 0.255    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.093    | 0.310    | 6.456    | 0.888        | 3.873    | 0.254    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.094    | 0.307    | 6.502    | 0.885        | 3.859    | 0.257    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.099    | 0.294    | 6.665    | 0.884        | 3.880    | 0.271    | 0.001    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.094    | 0.309    | 6.480    | 0.884        | 3.860    | 0.256    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.094    | 0.309    | 6.486    | 0.882        | 3.853    | 0.256    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.095    | 0.306    | 6.519    | 0.883        | 3.878    | 0.259    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.094    | 0.309    | 6.481    | 0.885        | 3.870    | 0.256    | 0.000    |
| 12.5 | 160 | 6.403 | 75-140       | 0.029    | 0.094    | 0.308    | 6.505    | 0.881        | 3.855    | 0.258    | 0.000    |
|      |     |       | Mean         | 0.029    | 0.141    | 0.226    | 9.384    | 0.715        | 3.870    | 0.582    | 0.000    |
|      |     |       | Std.<br>Dev. | 4.19E-04 | 4.59E-02 | 7.90E-02 | 2.82E+00 | 1.66E-<br>01 | 2.75E-02 | 3.18E-01 | 1.14E-04 |

|                 | PI                  |                  |        |                         |                        |                                |                  |                   |                           |                           |                   |
|-----------------|---------------------|------------------|--------|-------------------------|------------------------|--------------------------------|------------------|-------------------|---------------------------|---------------------------|-------------------|
| Output<br>power | Measurement<br>Time | Sensor<br>Radius | Points | Thermal<br>Conductivity | Thermal<br>Diffusivity | Volumetric<br>Specific<br>Heat | Probing<br>Depth | Temp.<br>Increase | Total<br>Temp<br>Increase | Total to<br>Char.<br>Time | Mean<br>Deviation |

| [mW]  | [s]    | [mm]  |                  | [Wm-1K-1] | [mm2s-1] | [MJm-3K-<br>1] | [mm]  | [K]   | [K]   |       | [K]   |
|-------|--------|-------|------------------|-----------|----------|----------------|-------|-------|-------|-------|-------|
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.032    | 0.373          | 1.594 | 1.320 | 8.741 | 0.055 | 0.000 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.033    | 0.366          | 1.617 | 1.319 | 8.772 | 0.056 | 0.001 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.031    | 0.380          | 1.574 | 1.316 | 8.773 | 0.053 | 0.000 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.030    | 0.385          | 1.556 | 1.317 | 8.754 | 0.052 | 0.000 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.030    | 0.386          | 1.558 | 1.348 | 8.749 | 0.052 | 0.001 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.030    | 0.387          | 1.543 | 1.317 | 8.743 | 0.051 | 0.001 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.011     | 0.028    | 0.409          | 1.484 | 1.323 | 8.794 | 0.047 | 0.001 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.011     | 0.027    | 0.417          | 1.462 | 1.317 | 8.775 | 0.046 | 0.000 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.011     | 0.029    | 0.400          | 1.510 | 1.318 | 8.772 | 0.049 | 0.001 |
| 8.000 | 20.000 | 3.189 | (125-<br>200,tc) | 0.012     | 0.030    | 0.387          | 1.546 | 1.323 | 8.746 | 0.051 | 0.001 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.083    | 0.169          | 3.165 | 2.136 | 6.783 | 0.216 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.084    | 0.169          | 3.170 | 2.137 | 6.784 | 0.216 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.084    | 0.168          | 3.172 | 2.137 | 6.745 | 0.217 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.084    | 0.169          | 3.169 | 2.136 | 6.781 | 0.216 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.084    | 0.169          | 3.170 | 2.135 | 6.799 | 0.216 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.083    | 0.169          | 3.160 | 2.135 | 6.766 | 0.215 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014     | 0.083    | 0.170          | 3.157 | 2.135 | 6.788 | 0.214 | 0.003 |

| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014 | 0.084 | 0.168 | 3.178 | 2.138 | 6.772 | 0.217 | 0.003 |
|-------|--------|-------|------------------|-------|-------|-------|-------|-------|-------|-------|-------|
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014 | 0.083 | 0.170 | 3.155 | 2.135 | 6.777 | 0.214 | 0.003 |
| 5.000 | 40.000 | 3.189 | (50-150)         | 0.014 | 0.091 | 0.158 | 3.306 | 2.130 | 6.776 | 0.235 | 0.003 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.077 | 0.183 | 2.486 | 0.616 | 3.777 | 0.133 | 0.001 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.078 | 0.181 | 2.505 | 0.621 | 3.769 | 0.135 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.074 | 0.189 | 2.435 | 0.625 | 3.747 | 0.128 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.070 | 0.196 | 2.371 | 0.623 | 3.794 | 0.121 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.069 | 0.199 | 2.348 | 0.619 | 3.812 | 0.119 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.077 | 0.183 | 2.479 | 0.621 | 3.802 | 0.132 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.073 | 0.191 | 2.413 | 0.628 | 3.770 | 0.125 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.074 | 0.189 | 2.429 | 0.622 | 3.804 | 0.127 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.079 | 0.180 | 2.511 | 0.617 | 3.814 | 0.136 | 0.000 |
| 3.500 | 20.000 | 3.189 | (125-<br>200,tc) | 0.014 | 0.068 | 0.200 | 2.334 | 0.624 | 3.801 | 0.117 | 0.001 |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015 | 0.075 | 0.199 | 3.461 | 0.455 | 3.141 | 0.258 | 0.001 |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015 | 0.077 | 0.194 | 3.511 | 0.452 | 3.133 | 0.265 | 0.001 |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015 | 0.073 | 0.204 | 3.411 | 0.458 | 3.154 | 0.250 | 0.001 |

| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.079    | 0.189    | 3.566    | 0.452        | 3.140    | 0.274    | 0.000    |
|-------|--------|-------|------------------|----------|----------|----------|----------|--------------|----------|----------|----------|
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.082    | 0.183    | 3.623    | 0.458        | 3.168    | 0.283    | 0.000    |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.073    | 0.203    | 3.418    | 0.457        | 3.130    | 0.251    | 0.000    |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.082    | 0.182    | 3.631    | 0.458        | 3.135    | 0.284    | 0.001    |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.082    | 0.183    | 3.620    | 0.460        | 3.138    | 0.282    | 0.001    |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.082    | 0.182    | 3.629    | 0.451        | 3.140    | 0.284    | 0.001    |
| 2.300 | 40.000 | 3.189 | (125-<br>200,tc) | 0.015    | 0.087    | 0.173    | 3.730    | 0.455        | 3.160    | 0.299    | 0.001    |
|       |        |       | Mean             | 0.014    | 0.067    | 0.234    | 2.679    | 1.134        | 5.618    | 0.167    | 0.001    |
|       |        |       | Std. Dev.        | 1.29E-03 | 2.22E-02 | 9.17E-02 | 7.83E-01 | 6.72E-<br>01 | 2.30E+00 | 8.63E-02 | 9.43E-04 |

|                |               | Polystyrene (X        | XPS)            |                      |
|----------------|---------------|-----------------------|-----------------|----------------------|
| Time           | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp. Increase |
| 160            | 427           | 1.59                  | 1.69            | 1.89                 |
| 160            | 430           | 1.59                  | 1.69            | 1.88                 |
| 160            | 468           | 1.59                  | 1.68            | 1.89                 |
| 160            | 439           | 1.59                  | 1.69            | 1.88                 |
| 160            | 433           | 1.59                  | 1.69            | 1.89                 |
| 160            | 454           | 1.59                  | 1.68            | 1.88                 |
| 160            | 452           | 1.59                  | 1.68            | 1.89                 |
| 160            | 465           | 1.59                  | 1.68            | 1.88                 |
| 160            | 465           | 1.59                  | 1.68            | 1.88                 |
| 160            | 471           | 1.59                  | 1.68            | 1.89                 |
| 160 [Average]  | 451           | 1.588                 | 1.68            | 1.89                 |
| 160 [Std. Dev] | 16.91         | 2.34E-16              | 1.57E-03        | 4.78E-03             |

| RTV-655 |               |                       |                 |                         |  |  |  |  |  |  |
|---------|---------------|-----------------------|-----------------|-------------------------|--|--|--|--|--|--|
| Time    | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |  |  |
| 40      | 164           | 0.32                  | 0.43            | 1.66                    |  |  |  |  |  |  |
| 80      | 359           | 0.57                  | 0.43            | 1.65                    |  |  |  |  |  |  |
| 160     | 512           | 1.21                  | 0.77            | 1.66                    |  |  |  |  |  |  |

| 160            | 517     | 1.21     | 0.90     | 1.93     |
|----------------|---------|----------|----------|----------|
| 160            | 522     | 1.21     | 0.98     | 2.10     |
| 160            | 523     | 1.21     | 0.98     | 2.09     |
| 160            | 524     | 1.21     | 0.97     | 2.10     |
| 160            | 525     | 1.21     | 0.97     | 2.09     |
| 160            | 526     | 1.21     | 0.97     | 2.09     |
| 160            | 532     | 1.21     | 1.13     | 2.44     |
| 320            | 656     | 0.99     | 0.62     | 1.75     |
| 320            | 682     | 1.42     | 1.03     | 2.01     |
| 320            | 686     | 1.42     | 1.23     | 2.39     |
| 320            | 689     | 1.42     | 1.23     | 2.39     |
| 320            | 691     | 1.42     | 1.22     | 2.39     |
| 320            | 694     | 1.42     | 1.22     | 2.38     |
| 320            | 694     | 1.42     | 1.22     | 2.38     |
| 40 [Average]   | 164     | 0.32     | 0.43     | 1.66     |
| 40 [Std. Dev]  | #DIV/0! | #DIV/0!  | #DIV/0!  | #DIV/0!  |
| 80 [Average]   | 359     | 0.57     | 0.43     | 1.65     |
| 80 [Std. Dev]  | #DIV/0! | #DIV/0!  | #DIV/0!  | #DIV/0!  |
| 160 [Average]  | 523     | 1.21     | 0.96     | 2.06     |
| 160 [Std. Dev] | 6.12    | 0.00E+00 | 9.97E-02 | 2.15E-01 |
| 320 [Average]  | 685     | 1.36     | 1.11     | 2.24     |
| 320 [Std. Dev] | 13.58   | 1.62E-01 | 2.27E-01 | 2.59E-01 |

| VR17 |  |
|------|--|

| Time           | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |
|----------------|---------------|-----------------------|-----------------|-------------------------|
| 160            | 558           | 1.21                  | 0.96            | 1.7234                  |
| 160            | 474           | 1.21                  | 1.05            | 2.0700                  |
| 160            | 470           | 1.21                  | 1.05            | 2.0822                  |
| 160            | 469           | 1.21                  | 1.06            | 2.0816                  |
| 160            | 472           | 1.21                  | 1.05            | 2.0706                  |
| 160            | 469           | 1.21                  | 1.05            | 2.0721                  |
| 160            | 470           | 1.21                  | 1.05            | 2.0777                  |
| 160            | 471           | 1.21                  | 1.05            | 2.0750                  |
| 160            | 472           | 1.21                  | 1.05            | 2.0735                  |
| 160            | 471           | 1.21                  | 1.05            | 2.0746                  |
| 160 [Average]  | 480           | 1.21                  | 1.04            | 2.04                    |
| 160 [Std. Dev] | 27.56         | 0.00E+00              | 2.83E-02        | 1.11E-01                |

| VR34 |               |                       |                 |                         |  |  |  |  |  |
|------|---------------|-----------------------|-----------------|-------------------------|--|--|--|--|--|
| Time | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |  |  |  |  |  |
| 160  | 504           | 1.06                  | 0.87            | 2.2311                  |  |  |  |  |  |
| 160  | 467           | 1.21                  | 1.11            | 2.3212                  |  |  |  |  |  |
| 160  | 444           | 1.06                  | 0.92            | 2.3512                  |  |  |  |  |  |
| 160  | 445           | 1.06                  | 0.92            | 2.3516                  |  |  |  |  |  |
| 160  | 443           | 1.06                  | 0.92            | 2.3694                  |  |  |  |  |  |
| 160  | 443           | 1.06                  | 0.92            | 2.3709                  |  |  |  |  |  |
| 160  | 444           | 1.06                  | 0.92            | 2.3734                  |  |  |  |  |  |
| 160  | 443           | 1.06                  | 0.92            | 2.3745                  |  |  |  |  |  |

| 160            | 445   | 1.06     | 0.92     | 2.3759   |
|----------------|-------|----------|----------|----------|
| 160            | 443   | 1.06     | 0.92     | 2.3803   |
| 160            | 443   | 1.06     | 0.92     | 2.3812   |
| 320            | 462   | 0.60     | 0.51     | 2.0918   |
| 320            | 443   | 0.60     | 0.60     | 2.4561   |
| 320            | 443   | 0.60     | 0.60     | 2.4584   |
| 320            | 442   | 0.60     | 0.61     | 2.4683   |
| 160 [Average]  | 451   | 1.07     | 0.93     | 2.35     |
| 160 [Std. Dev] | 18.82 | 4.55E-02 | 5.92E-02 | 4.41E-02 |
| 320 [Average]  | 448   | 0.60     | 0.58     | 2.37     |
| 320 [Std. Dev] | 9.80  | 0.00E+00 | 4.56E-02 | 1.85E-01 |

| VR51 |               |                       |                 |                         |
|------|---------------|-----------------------|-----------------|-------------------------|
| Time | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |
| 160  | 649           | 0.76                  | 0.568057        | 1.853                   |
| 160  | 646           | 0.76                  | 0.568348        | 1.855                   |
| 160  | 648           | 0.76                  | 0.568189        | 1.861                   |
| 160  | 647           | 0.76                  | 0.568254        | 1.866                   |
| 160  | 680           | 1.05                  | 1.06            | 3.009                   |
| 160  | 681           | 1.05                  | 1.06            | 3.013                   |
| 160  | 683           | 1.05                  | 1.06            | 3.016                   |
| 160  | 680           | 1.05                  | 1.06            | 3.021                   |
| 320  | 828           | 0.85                  | 0.63            | 1.837                   |
| 320  | 719           | 0.85                  | 0.68            | 1.937                   |
| 320  | 716           | 0.85                  | 0.68            | 1.939                   |

| 320 | 720  | 0.85 | 0.68 | 1.945 |
|-----|------|------|------|-------|
| 320 | 716  | 0.85 | 0.68 | 1.951 |
| 320 | 749  | 1.35 | 1.32 | 3.397 |
| 320 | 750  | 1.35 | 1.32 | 3.399 |
| 320 | 748  | 1.35 | 1.32 | 3.403 |
| 320 | 747  | 1.35 | 1.32 | 3.403 |
| 320 | 739  | 1.35 | 1.33 | 3.427 |
| 320 | 1702 | 3.46 | 1.45 | 5.801 |
| 320 | 1939 | 3.46 | 1.45 | 6.189 |
| 320 | 3010 | 3.46 | 1.15 | 6.190 |
| 320 | 2750 | 3.46 | 1.24 | 6.332 |
| 320 | 2832 | 3.46 | 1.28 | 6.632 |
| 320 | 1947 | 3.46 | 1.57 | 6.747 |
| 320 | 3227 | 3.46 | 1.24 | 6.886 |
| 320 | 3102 | 3.46 | 1.28 | 6.980 |
| 320 | 2972 | 3.46 | 1.32 | 7.054 |
| 320 | 2862 | 3.46 | 1.37 | 7.129 |
| 320 | 3263 | 3.46 | 1.31 | 7.353 |
| 640 | 767  | 1.33 | 1.35 | 3.331 |
| 640 | 742  | 1.33 | 1.37 | 3.373 |
| 640 | 7474 | 1.47 | 0.65 | 3.835 |
| 640 | 3    | 1.47 | 1.55 | 4.541 |
| 640 | -4   | 1.47 | 1.55 | 4.559 |
| 640 | -11  | 1.47 | 1.62 | 4.744 |
| 640 | -43  | 1.47 | 2.01 | 5.831 |
| 640 | 3385 | 1.47 | 1.35 | 5.836 |

| 640            | -115    | 1.47     | 2.32     | 6.668    |
|----------------|---------|----------|----------|----------|
| 640            | -164    | 1.47     | 2.63     | 7.500    |
| 640            | 889     | 1.47     | 2.53     | 8.263    |
| 640            | 49      | 1.47     | 2.99     | 8.745    |
| 160 [Average]  | 664     | 0.90     | 0.82     | 2.44     |
| 160 [Std. Dev] | 18.09   | 1.58E-01 | 2.65E-01 | 6.18E-01 |
| 320 [Average]  | 1764    | 2.34     | 1.17     | 4.76     |
| 320 [Std. Dev] | 1072.49 | 1.22E+00 | 2.99E-01 | 2.14E+00 |
| 640 [Average]  | 1081    | 1.45     | 1.83     | 5.60     |
| 640 [Std. Dev] | 2241.49 | 5.69E-02 | 6.76E-01 | 1.86E+00 |

| PI-Sheets |               |                       |                 |                         |
|-----------|---------------|-----------------------|-----------------|-------------------------|
| Time      | Specific Heat | Reference<br>Increase | Sample Increase | Total Temp.<br>Increase |
| 40        | 73            | 0.20                  | 0.21            | 1.2858                  |
| 80        | 1233          | 0.71                  | 0.68            | 2.0636                  |
| 80        | 1250          | 0.71                  | 0.68            | 2.0667                  |
| 80        | 1257          | 0.71                  | 0.68            | 2.0668                  |
| 80        | 1247          | 0.71                  | 0.68            | 2.0682                  |
| 80        | 1232          | 0.71                  | 0.68            | 2.0794                  |
| 160       | 547           | 0.37                  | 0.38            | 2.2701                  |
| 160       | 317           | 0.20                  | 0.22            | 2.2937                  |
| 160       | 306           | 0.20                  | 0.22            | 2.2943                  |
| 160       | 555           | 0.37                  | 0.38            | 2.2972                  |
| 160       | 308           | 0.20                  | 0.22            | 2.2987                  |

| 160           | 315    | 0.20     | 0.22     | 2.3009   |
|---------------|--------|----------|----------|----------|
| 160           | 302    | 0.20     | 0.22     | 2.3047   |
| 320           | 1108   | 0.52     | 0.50     | 2.8701   |
| 320           | 1114   | 0.52     | 0.50     | 2.8744   |
| 320           | 1117   | 0.52     | 0.50     | 2.8749   |
| 320           | 1110   | 0.52     | 0.50     | 2.8765   |
| 320           | 1098   | 0.52     | 0.50     | 2.8856   |
| 320           | 1112   | 0.52     | 0.54     | 3.1061   |
| 320           | 2220   | 1.35     | 1.26     | 3.2953   |
| 320           | 2186   | 1.35     | 1.27     | 3.2978   |
| 320           | 2178   | 1.35     | 1.27     | 3.3077   |
| 320           | 2165   | 1.35     | 1.27     | 3.3146   |
| 320           | 2167   | 1.35     | 1.27     | 3.3164   |
| 320           | 2074   | 1.35     | 2.37     | 6.1695   |
| 320           | 2089   | 1.35     | 2.37     | 6.1719   |
| 320           | 2075   | 1.35     | 2.37     | 6.1756   |
| 320           | 2056   | 1.35     | 2.37     | 6.1808   |
| 320           | 2081   | 1.35     | 2.37     | 6.1970   |
| 640           | 2542   | 1.33     | 1.17     | 3.1142   |
| 640           | 2473   | 1.33     | 1.17     | 3.1305   |
| 640           | 2507   | 1.33     | 1.17     | 3.1307   |
| 640           | 2455   | 1.33     | 1.17     | 3.1325   |
| 640           | 2484   | 1.33     | 1.17     | 3.1338   |
| 40 [Average]  | 977    | 0.59     | 0.57     | 1.99     |
| 40 [Std. Dev] | 475.91 | 2.14E-01 | 1.93E-01 | 3.18E-01 |
| 80 [Average]  | 1575   | 0.91     | 1.06     | 3.49     |

| 80 [Std. Dev]  | 824.05 | 5.01E-01 | 7.51E-01 | 1.35E+00 |
|----------------|--------|----------|----------|----------|
| 160 [Average]  | 379    | 0.25     | 0.27     | 2.29     |
| 160 [Std. Dev] | 117.91 | 8.34E-02 | 7.68E-02 | 1.13E-02 |
| 320 [Average]  | 1747   | 1.04     | 1.33     | 4.06     |
| 320 [Std. Dev] | 511.76 | 4.18E-01 | 7.94E-01 | 1.49E+00 |
| 640 [Average]  | 2492   | 1.33     | 1.17     | 3.13     |
| 640 [Std. Dev] | 33.76  | 0.00E+00 | 3.00E-03 | 8.03E-03 |

## APPENDIX B

## THERMAL CONDUCTIVITY MEASUREMENT GUIDE

- 1. Unplug all sensors from TPS 1500, then turn on computer and TPS 1500.
- 2. Secure samples and sensor with sample holder, then plug sensor into TPS 1500.
- 3. Open software "Hot Disk Thermal Constants Analyzer 7.3" on the desktop.
- 4. For Thermal Conductivity Measurements Select ISOTROPIC only then choose the BULK (TYPE 1) measurement.
- 5. Type sample name and available probing depth in top section of settings.
- 6. Choose sensor design, insulation, and cable/holder under second section.
- 7. Set temperature source to USER VALUE, then input the temperature value.
- 8. Assign heating power and measurement time according to the values listed in the table below.
- 9. Wait at least one hour to allow sample temperature to stabilize.
- 10. Click on green start button, then confirm the settings to initiate the measurement.
- 11. Wait for measurement to finish.
- 12. Inspect temperature **drift** graph to ensure the sample was at equilibrium. If the graph exhibits a slope, repeat the measurement after a minimum of thirty minutes.

- 13. Click **Calculate**, then choose desired data range and measurement options. Use data points 15-200 for first calculations. If the total temperature increase is not between 2-7K, repeat measurement after a minimum of thirty minutes.
- 14. Optimize the data range to achieve acceptable values for **total to characteristic time**,  $\Theta$ ; **probing depth**; and **mean deviation**. (Note: 0.333 < $\Theta$  <1; probing depth < available probing depth; 1x10<sup>-3</sup> > mean deviation)
- 15. Click **Export Results** and save the Excel file. Warning: do not close or navigate out of the exporting file or the Excel file will be damaged.